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Topics of the Month

Rearmament and chemical engineering

THERE is much loose thinking about the size of the demand which is being made upon our industrial resources by the present rearmament programme. Misunderstanding and anxiety is fostered by the remarks of certain politicians who talk about the country being on a war footing. Compared with our effort in the last war the present programme is quite modest. The immediate past president of the Federation of British Industries, Sir Robert Sinclair, for instance, estimates that at its peak the volume of defence work will represent about 20% of current total output of the engineering industry, compared with 80% in the war. If it were possible to continue to increase production the effects of rearmament upon supplies for the home and export markets might be even less than is indicated by this figure. Unfortunately, shortages of raw materials make it impossible to hope for higher production so that the effects will be more severe than this figure adumbrates.

What of the effects of rearmament upon the chemical plant industry? This question is examined in the annual report of the British Chemical Plant Manufacturers' Association which appeared last month. The Association wants the industry to continue with its normal production without having to take on defence projects not of a chemical engineering nature which would upset production, slow down supplies to the home chemical industry and seriously weaken the chemical plant export drive. Bearing in mind the supreme importance of the chemical industry in both peace and war there is every justification for the plant manufacturers wishing to continue to serve it without hindrance.

State company to develop Norway's minerals

THE Norwegian Government will shortly be starting work on mining niobium—a metal which on account of its very high resistance to heat is much in demand for jet engines and other equipment in which the metals are exposed to severe strain at high temperatures. Largely with this object in view, a Bill for the establishment of a State mining company has been put forward, and last month it passed its final stage in Parliament. The new company will mainly be a co-ordinating organ to take over and run seven mining companies in which the State already has interests or a share majority. In addition, there are a large number of mineral ore deposits in which the State has rights but which have not yet been developed.

Among the most important of these deposits is that at Holla, near Skien in Telemark, where it is estimated that a rock, named 'Soevit' by the Norwegian geologists who discovered it, contains large quantities of niobium. The deposits were investigated first in 1910 by the Notodden Saltpetre Factory, but the Soevit was not found suitable for saltpetre production. Later, during the occupation, the Germans spent large sums on research as they needed niobium for V.1 and V.2 projectiles. But they did not complete the work before the end of the war.

E.C.A. representatives in Norway have now urged that the deposits should be developed for the niobium, and the Norwegian Defence Ministry's Research Institute has already worked on experimental methods of obtaining pure metallic niobium. By-products will be potash and phosphates.

As far as is known, niobium is to be found in large quan-

ties only in Nigeria, and on the Kola peninsula in the Soviet Union. The price per kilogramme is over \$500 and it is estimated that the Norwegian Soevit deposits should yield about 130 tons of niobium concentrate annually.

Speaking in the Parliamentary debate, the Norwegian Commerce Minister pointed out that a State mining company was necessary, particularly for administering the niobium deposits which were now the object of international economic co-operation. This matter could not be left to private interests, he said. The research and development of the mineral could only be achieved with help from abroad, and Norway was to receive dollars and technical assistance for the purpose.

The new State mining company will not constitute a mining monopoly, as there are several private companies engaged in mining ores in various parts of Norway. The new company will merely take over those companies in which the State already has rights or interests.

New method of superphosphate manufacture conserves sulphuric acid

THE fertiliser industry is the biggest single consumer of sulphuric acid in this country. It has doubled its consumption in recent years and now uses well over one-third of the total supplies available to British industry. A large part of its consumption—about 470,000 tons—goes to make superphosphates.

Any process for making superphosphates which would economise in the use of the acid is particularly important in the present world shortage of sulphur.

Superphosphate is made by the action of sulphuric acid on phosphate rock. The product is a mixture of mono-calcium phosphate and calcium sulphate. The increasing problem of sulphur supply has led the Chemical Research Laboratory, D.S.I.R., to examine alternative methods of making phosphate fertilisers. The object of the investigation was either to reduce the amount of sulphuric acid necessary or to eliminate it entirely. One of the important considerations which had to be taken into account was the necessity of developing a method which could use the existing plant designed for the sulphuric acid process without major alteration.

The most attractive possibility was to use nitric acid instead of sulphuric, for nitric acid does not require imported materials for its manufacture. The reaction of nitric acid on phosphate rock is completed in a relatively short time. As well as calcium phosphate, calcium nitrate is formed as a by-product, and calcium nitrate is itself a valuable fertiliser. The disadvantage is that it absorbs moisture very readily. The fertiliser therefore cakes badly in storage and is of no use to the farmer. Removing the nitrate would take too long and in any case would increase the cost too much. Even then some use would have to be found for the nitrate by converting it into some form of fertiliser less sensitive to moisture.

The successful method developed by the Chemical Research Laboratory consists in treating rock phosphate with a mixture of nitric acid and sulphuric acid, in almost the same way as the original process using sulphuric acid alone. The proportion of nitric to sulphuric acid is about 4 to 3. The method is successful because calcium sulphate is formed as well as calcium nitrate. Calcium sulphate is inert and will not pick up any moisture, no matter how damp the atmosphere. Since it also has the property of coating the grains of calcium nitrate, it protects this also from becoming damp.

The process gives a product which is relatively non-caking and will keep reasonably dry in the normal British climate. Experiments have also been carried out to produce a 'complete' fertiliser by adding potassium chloride, the chief source of potash. A P-N-K fertiliser containing 10% of potash was prepared. This material was more sensitive to moisture, but its laboratory tests were very severe and it is likely to prove a reasonably good product in practice.

Dutch chemical expansion

THE Dutch chemical industry, which has undergone considerable expansion since the war, achieved results last year that showed an even better performance than in 1949. Production of most major items with the notable exception of phosphate fertilisers climbed well above the previous year's figures.

The following index figures illustrate progress made over the past three years (1938 = 100):

	1948	1949	1950
Benzole products	166	191	215
Sulphuric acid	78	83	91
Phosphate fertilisers	164	173	129
Nitrogen fertilisers	82	88	149
Raw materials for paints (1939 = 100)	68	83	100
Paints (1939 = 100)	—	105	115
Organic acids (1948 = 100)	100	—	170

Optimism about the future is, however, tempered by the knowledge that the absence of German competition in many fields after the war made the industry's task very much easier. Soaring prices of raw materials are now making progress more and more difficult.

Various projects now being carried out by the Dutch State mines will be of great importance to Dutch chemical manufacturing. A new phenol plant, expected to be ready in 1952, will be of great value to the rayon and plastics industries. The most modern coking plant in Europe is now under construction at Limburg and this will produce several important chemical products, mostly for export. The State mines will also expand production of nitrogen. Financial aid for these plans is being given by the Reconstruction Bank. In other directions, too, activity is being stepped up, e.g. dyestuffs and pharmaceutical products.

Many millions of guilders are being spent to develop and improve processes and products so that Holland's chemical manufacturers may keep pace with their overseas competitors.

Development of Canadian titanium discoveries

ALTHOUGH tremendous developments and multiple uses are forecast for titanium metal, as a result of the immense ore discoveries on the North Shore of the St. Lawrence in Quebec Province, its immediate and vital importance is based on the market for titanium dioxide as a pigment.

A recent survey, points out that almost all the titanium dioxide that is being produced in Canada will find plenty of demand for use as a pigment, chiefly in the production of paints, and that the whole vast project under way in Quebec is based on the market for titanium dioxide alone in its present and expanding use as a pigment. If an economical method of producing the pure metal is found, and titanium takes its place as a structural material, it is likely that smelting facilities at Sorel would be enormously expanded.

As a pigment, titanium reveals additional remarkable properties. Owing to its high opacity and brightness, it is now considered to be the top quality for making white paints. It is used in almost all makes of white enamels and

paints for inside use, and replaces some of the pigments in most high-quality ready-mixed house paints, while special grades are used in white and tinted outside enamel and paints.

An estimated 75% of titanium dioxide pigments produced are consumed in the paint, enamel and lacquer industries, but it is also used in a score of other applications in the paper industry; as a delustrant, brightener and stiffener in textiles; for making white and light-coloured leather; in plastics, floor coverings and many others.

Its growth in these fields alone, since a method of separating the dioxide from ilmenite was developed before World War I and commercial production got under way in the 1920s, has been astounding. In the U.S.A., production of titanium pigment climbed from 4,000 tons in 1925 to 275,000 tons in 1946. Titanium, both in the form of the metal and its derived products, is likely to increase rapidly its fields of application.

South Africa's petrol-from-coal plant

WHEN present plans are translated into production South Africa will possess a very considerable liquid fuels industry. Last month we described the Vacuum Oil scheme for a large oil refinery near Durban. To this project must be added another—the South African Coal, Oil & Gas Corporation's plan for a plant to produce 60,000,000 gal. of petrol p.a. from coal. As we reported in a news item last December (p. 560) the plant will also produce diesel oil and certain quantities of gas.

More details of the proposed plant have now been sent us by the M. W. Kellogg Co., who have secured the contract to build the plant. The scheme envisaged falls into three parts: the opening of virgin coal deposits to supply raw material; the construction of above-ground gasification facilities to transform coal into synthesis gas; and the complete synthesis plant, heart of the project, to produce liquid hydrocarbons. Kellogg's *Synthol* process is to be utilised in the synthesis step. It differs materially from other processes used in synthetic petrol plants. Recent research on the process has produced a new type of reactor to handle powdered catalyst and new fundamental operating conditions.

If the scheme is successful it will go far to make up for South Africa's lack of major indigenous petroleum deposits by utilising her tremendous reserves of low cost coal for liquid fuel manufacture. High octane petrol processes such as catalytic polymerisation will form part of the plant facilities. By-products will be alcohols and oxygenated chemicals suitable for solvents and other uses.

Timber and pulp

EGO GLESINGER'S remarkable book, 'The Coming Age of Wood' (see INTERNATIONAL CHEMICAL ENGINEERING, April 1950, p. 157), vigorously presented the case for integrating the timber and wood-pulp industries of North America. Glesinger suggested that the wood wastes of the timber or lumber industry could be turned into three times as much pulp as the United States consumes annually; yet whole trees were felled and pulped and huge tonnages of pulp were imported. In Northern Europe these two major wood-using industries had not remained so completely isolated, so that much less wood suitable for building timber was pulped and much less wastage from board-cutting operations was burnt or left to decay.

With so much of the civilised world feeling the increasing pressure of paper shortage, it is heartening to learn that this

type of integration is taking place in the Canadian forests of British Columbia. Substantial deals have been made between major timber and pulp companies and some of the largest timber firms are now associated with pulp factories. Glesinger expressed the view that a national board or forest products corporation would be needed to impose integration upon these separate industries in North America. The beginning of this process in west Canada, however, is being stimulated by the high world price for wood pulp—that is to say, the laws of supply and demand, operating freely, have made the two forest-consuming industries come together even to the extent in some cases of capital mergers.

When the huge demands made by the United States upon the world's pulp production are considered, it is to be hoped that integration between sawmills and pulp factories will take place south of the 49th parallel. There are 25,000 separate lumber and pulp mills in the U.S. Integration of about 80% of these mills would reduce the annual consumption of trees substantially and make the U.S. a very small importer of kraft pulp and newsprint.

Lignin as a rubber reinforcement

In these notes in our February 1951 issue we reported that the Americans had discovered that a paper mill waste product, *p*-cymene, could be used to make *p*- α -methyl styrene which may replace or supplement styrene in the manufacture of synthetic rubber. Now comes news that yet another paper industry waste product, lignin, is a potentially valuable raw material in rubber manufacture. At the spring meeting of the American Chemical Society's Division of Rubber Chemistry, three chemists of the West Virginia Pulp & Paper Co. reported that lignin is an excellent reinforcing agent for both natural and synthetic rubber. Added to natural rubber in proportions of 50 to 100 lb./100 lb. rubber, lignin was found to give higher tensile strengths than any other pigment at the same volumes, including carbon black.

In the nitrile type of synthetic rubber, lignin also gave higher tensile strengths than any other pigment, according to these workers, who tested all the major types of rubber reinforcing agents, including whiting, magnesium carbonate, calcium carbonate, coated calcium carbonate, calcium silicate and three kinds of carbon black.

A gummy material present in huge quantities in pulp mill waste liquors, lignin had previously been shown to be an outstanding reinforcing agent for the general purpose synthetic rubber, GR-S. Used in water solution, lignin is mixed with the rubber latex and the mixture is solidified by a process called co-precipitation. With minor changes, this process could be carried out in the existing equipment of latex master-batching plants.

The combination of properties obtainable with lignin when co-precipitated with natural, nitrile or neoprene rubbers as well as with GR-S is novel. In addition to tensile strengths which are at least of the same order as those obtained with the carbon blacks and in certain cases superior, good abrasion resistance and tear resistance result. In GR-S, nitrile, and natural rubber, high elongation and low modulus are combined with the high tensile strength and high hardness.

Although co-precipitated lignin is itself dark in colour, it has a low tinting power. Except for white and pastel shades, colours are readily obtainable by blending with other pigments, retaining the strength values derivable from the lignin.

International coal classification

COAL samples from Belgium, France, Holland, Poland, U.S.A., W. Germany and U.K. were recently analysed in all the above countries and in Italy and Sweden so that results could be compared and a tentative classification of European coals established. The Coal Committee of the United Nations Economic Commission for Europe has been working for some time on the establishment of an international and commercially operable system of coal classification and coal nomenclature in order to eliminate much of the confusion and practical inconvenience caused at present by the absence of such a system. These tests recently carried out for this committee have permitted comparison to be made of the different methods of determining moisture, ash, carbon and volatile matter content, calorific values and caking properties employed in the different countries.

In one case the committee agreed to the Dutch proposal for the testing of British caking coals in continental laboratories to determine whether the dilatometer test largely used on the Continent produced results similar enough to those of the Gray King test, used in the U.K., so that both tests could be employed to designate the characteristics to be used in the international classification system.

Some new tests have now been scheduled and it is hoped the results and some further questions can be considered at a further meeting this summer so that an agreed system for classifying European coals can be established.

New method of producing ethyl alcohol

WHILE investigating a method for converting an alcohol to the primary alcohol containing one carbon atom more than the original (homologation), under conditions resembling those employed in the 'oxo' reaction, U.S. Bureau of Mines' workers found that methyl alcohol reacts with the synthesis gas used to give ethyl alcohol as the chief reaction product. This conversion presents an interesting new route to the alcohol which may have important theoretical and commercial implications.

In one experiment, 64.1 g. of methyl alcohol and 4 g. of dicobalt octacarbonyl $[\text{Co}(\text{CO})_4]_2$ were placed in a stainless steel autoclave and synthesis gas (H_2 : ICO) was added until the pressure reached 3,500 lb./sq.in. The autoclave was then heated with rocking to 185°C . within 65 min., the maximum pressure obtained being 5,100 lb./sq.in. at 181°C .

The temperature was kept at 183 to 185°C . for 4 hr., during which time the pressure dropped to 2,410 lb./sq.in. The reaction vessel was cooled to room temperature and then repressured to 3,000 lb./sq.in. with 1:1 synthesis gas. The temperature was raised to 185°C . once more (maximum pressure, 4,560 lb./sq.in. at 167°C .) and maintained for 4 hr., during which time the pressure dropped to 3,150 lb./sq.in. The total pressure drop corresponded to approximately 3.6 moles of gas, or 2.3 moles of gas per mole of methyl alcohol converted.

After cooling to room temperature overnight, gas samples were taken and analysis of the carefully fractionated mixture showed that 49 g. (76.4%) of the methyl alcohol had reacted to give the following products: methyl formate 2%, methyl acetate 9%, ethyl alcohol 38.8%, ethyl acetate 6.3%, propyl alcohol 4.7%, butyl alcohol 0.9%, methane 8.5% and propyl acetate 0.1%.

The absence of appreciable quantities of higher alcohols in the products from the reaction of methanol indicated that

ethanol would probably react sluggishly in the homologation reaction.

This was confirmed by experiment; a 4.1% yield of *n*-propanol and small quantities of butyl alcohols were secured after a long period of reaction. The variety and distribution of products obtained from methyl alcohol in the homologation reaction have certain similarities to the oxygenated products secured in the Fischer-Tropsch process, but the preponderance of ethanol and the relatively small amounts of methanol, *n*-propanol and butanols in the Fischer-Tropsch process products may be significant in comparison with the rapid conversion of methanol and the slow reaction of ethanol characteristic of the homologation reaction. According to the report of this work in *Science* (1951, 113, p. 206), the yield of ethanol from methanol could probably be increased by increasing the hydrogen: carbon monoxide ratio of the synthesis gas. Any salt of cobalt yielding the carbonyl under the conditions of the reaction will serve as a catalyst.

A hundred years ago

THE chemist is much concerned in a double centenary due to be celebrated in the world of photography. The first concerns collodion and the quieter peace-time applications of cellulose nitrate. For it was Frederick Scott Archer in 1851 who first applied the collodion process in photography, where practical directions for its use were concerned. He was challenged by De Gray, French painter who independently used collodion, it is true. And whenever collodion of 1851 is mentioned, the name of Robert Bingham crops up, for this quick worker improved the process in a matter of weeks and, on being sent by the British Government to photograph a Paris exhibition, he made 2,500 photographs in a short time, only made possible with collodion. Schönbein in 1846 had discovered gun-cotton; Louis Ménard, who invented collodion as a laboratory curiosity, worked with Domonte, and the two of them showed that, whereas gun-cotton remained undissolved in an alcohol-ether mixture, the less nitrated collodion was soluble—hence the possibility of use. Yet, while others like Pérouze, Abel, and Crum with Schönbein added to the chemist's studies of nitrocellulose, Scott Archer produced the first collodion negatives and modern photography became possible. Scott Archer followed this by his stripping process for preserving negatives without glass, by coating collodion films with rubber solution before stripping.

Although there is much more we could give, of Ménard as a Greek cynic philosopher walking through Paris with wooden shoes and straw peeping out, for example, the second point for commemoration concerns Daguerre's death in 1851. The 'Archerotype' was hardly known, compared with the 'Daguerrotype,' as so often happens in perpetuating a name. Daguerre worked with Niepce and light-sensitive silver salts resulted. Niepce first tried silver chloride on paper, and also tried etching plates and exposing them to iodine vapour. But the classical Daguerrotype came as a result of one of those accidental discoveries common to progress in science. For Daguerre silvered a copper plate, iodised it, and developed it after exposure with mercury vapour—all because he happened to put a plate in a cupboard containing an uncorked bottle of mercury. Like his countryman De Gray, Daguerre was painter rather than chemist, yet his work with silver iodide, his fixing with thiosulphate, and toning with gold chloride were striking achievements for an 'amateur.'

The Hydration of Lime

By J. Lomas

THE slaking of lump lime by hand on the site of its actual employment is rarely satisfactory, involving a good deal of labour, much loss of material and uncertain results. For this reason, the modern tendency is increasingly to market lime in a hydrated form, particularly for the building and constructional industries wherever a caustic or drying action is unnecessary and the lime need not be of hydraulic type. Farmers are also buying lime in the hydrated form, though a natural conservatism combined with an unduly high price has not yet resulted in the complete adoption of this type of material.

The mechanical hydration of lime involves in most instances three separate and distinct phases: In the first, the quicklime in lump form has to be crushed or ground to a reasonably uniform small size; in the second, this reduced material must be well mixed with water; and, in the third, the lime thus slaked has to be screened to final particle size or reduced to a fine powder in some other manner. In this article we shall take each of these stages and discuss it in detail.

Grinding

As regards the grinding of the quicklime, the type of plant used depends to a considerable extent on the purpose for which the ultimate product is required. For the building trade and the manufacture of chemical products, it is customary to employ either a ring roll mill, a ball mill or a type of hammer mill specially designed for the purpose. The degree of fineness attained is variable. If the product in advance of hydration is not required to be greater than $\frac{1}{2}$ in. or 1 in., a crusher of small size is employed, and the fine grinding mills are used later to reduce the product of the crusher. The main point is to secure uniformity of the quicklime in advance of slaking, as otherwise it will prove exceptionally difficult to obtain the required degree of consistency in the results. The old practice was to use hand labour, but this is rarely practised in up-to-date plants. The crusher used is often provided with a screen and hopper to screen out lumps of a diameter smaller than 2 in., so that only those above this size pass through the machine. Automatic feeders are sometimes used in conjunction

From the building and agricultural industries there is an increasing demand for lime in hydrated form. The hydration of lime involves three distinct operations: crushing of quicklime, slaking and, finally, screening to the desired particle size. These processes and the plant required are described in detail in this article.

with the crusher. Table 1 gives an indication of the capacity of a small crusher designed to produce quicklime to pieces of about $\frac{1}{2}$ in.

This preliminary crushing of the lime should on no account be eliminated, as it leads to pieces of unslaked material in the final product. Many manufacturers screen out anything below $\frac{1}{2}$ in. diam., and subject the material so obtained to a quite separate hydrating process. This has many advantages.

For a number of chemical purposes, the lime is brought to a considerable degree of fineness, but for normal reductions, a ball mill can be used in combination with an air separator. As an example of the output to

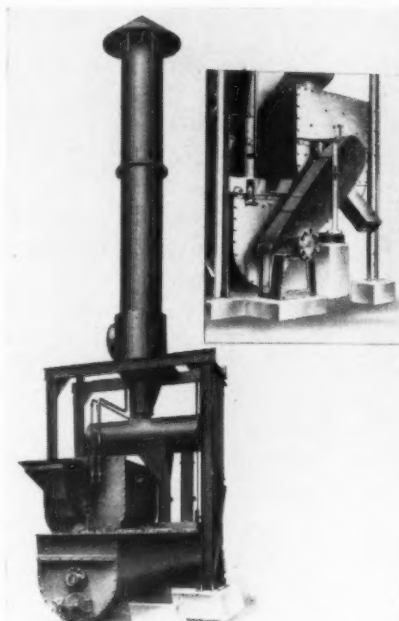
be expected from, for example, a mill 7×3 ft., with a size of feed of 90% through 100 mesh, we may take the estimated figure of 12.5 tons/hr., which would call for an h.p. of 100 for the mill and 35 for the auxiliary plant. Such a mill would have a ball tonnage of about 9, and would run at 20 r.p.m. To give a lime product of 99 to 99.5% through 200 mesh, such as is often required in the sugar-beet industry, a mill of rotary type operating on a lime of 95% CaO would produce an output of 5.4 tons/hr. This would necessitate a total h.p. of 110, or 20.3 h.p./ton.

There are conflicting points of view as to the necessity of grinding the quicklime to such fineness before hydration, and British practice tends to the opinion that it is better to crush down to not less than $\frac{1}{2}$ in. diam., and store the hydrated product in a bin for a few days, rather than fine-grind, without storing, which is said to be harmful as allowing unslaked lime to enter the bricks, plaster or paints in which the hydrated product is employed.

Slaking

We now come to the actual slaking of the crushed or ground lime. There is no one standard practice for this, the methods employed being mainly governed by the kind of system adopted. Before any particular plant is purchased, it is essential to remember that there must be adequate water supply, the rate of water addition must be correct and the method of addition efficient, and mixing must be carried out in the best possible manner. Temperature is most important, as the inevitable generation of heat during the slaking operation may result in the 'burning' of the lime, unless the heat is withdrawn rapidly, but not so rapidly as to cause interruption of the hydration process. Some control of temperature is achieved by modifying the stirring of the material, but thorough efficiency in this respect is only obtainable as a result of continual testing.

Mixing is normally achieved by means of a pan provided with agitators, somewhat on the same lines as a concrete mixer, though a horizontal-drum-type mixer may on occasion be preferred. The lime is either added to the water at one operation, or may first be mixed with an excess of water, and the rest of the lime added afterwards. It is essential that lime and water should be kept together long enough to ensure thorough hydration. Most limes can be slaked thoroughly in 24 hr., but some limes, especially those of magnesian type, slake very slowly, and must be given as long a period as possible. Otherwise, there is always a risk that the lime or a proportion of it may slake after it has



Schulthess lime hydrator. A general view from the feed end.

(This photograph and that on p. 214 by courtesy of Edgar Allen & Co., Ltd.)

TABLE 1

Hopper mouth, in.	Capacity, tons hr.	h.p.	r.p.m.	Pulley diam. and face, in.
13 x 18	2-6	6-10	300	24 x 8
20 x 30	8-15	15-20	250	32 x 12

been put into use, and this may set up expansion of dangerous type. This factor of choice of lime is important, since not every lime has the same slaking properties. Calcareous limes composed mainly or entirely of calcium oxide will prove most satisfactory, and if these are used, 100 lb. should yield 133 lb. of hydrated material, of which about 122 lb. will be true hydrated lime and the balance inert substance.

The quantity of water employed depends on the type of lime. For high-calcium limes, a greater amount of water is necessary than for limes rich in magnesia or clayey matter.

Types of hydrator

One of the most interesting hydrators is the Schulthess, shown on page 213. The particular advantage of this machine is that it can be employed without any previous breaking up of the lime. The crude lime can be fed into the hopper just as it comes from the kilns, and without further preparation. The product is a dry, fine powder, free from lumps. The dry slaking is obtained without excessive production of lime dust, so that comfortable operating conditions and a dust-free environment are secured. Slaking is rapid, and the plant is continuous in operation, all that is required being to keep the hopper well supplied with crude lime. The slaking water is automatically and continuously heated, and the lime dust contained in the steam is recovered, thus preventing loss of lime. The machine can be provided with mechanical feed, and calls for an unusually small motive power, approximately one-third that of other mechanical lime-slaking methods. The product, when sifted, is of great purity and, if protected from the air, can be kept for an almost unlimited time.

Table 2 shows the capacities of the various sizes of hydrator of this type, based on the use of high-calcium lime.

TABLE 2

	Size No.					
	1	2	3	4	5	6
Tons per hr. (average)	$\frac{1}{2}$	1-1 $\frac{1}{2}$	1 $\frac{1}{2}$ -2	2-3	3-4	5-7
Approx. h.p.	2	4	6	8	9	12

The machine comprises two chambers superimposed and suitably connected. The first chamber is short and contains a rotary perforated drum for the preliminary slaking of lump lime, the final slaking being carried out in the finishing chamber, which has a centre shaft with mixing and transporting paddles.

In operating this hydrator, the burnt lime from the kilns is fed into the preliminary slaking screen at the feed chute, and a water spray pipe is fitted internally. The machine is set in motion and the water turned on. Heat and steam generated reduce the crude lime to powder and small

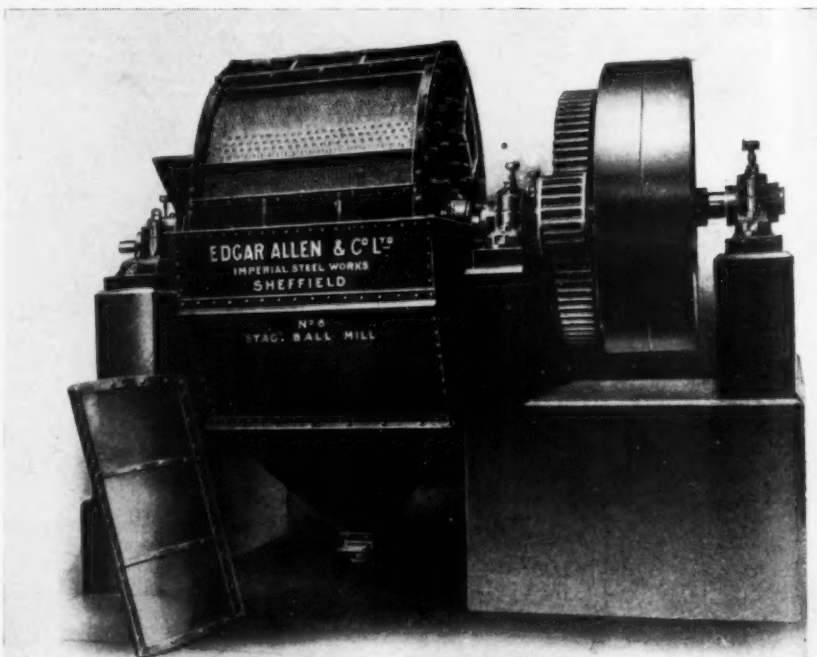
particles, which fall through the holes in the revolving screen, and are propelled by a spiral in the annular space between the outer periphery of the screen and the casing, falling thence by gravity down a chute into the finishing chamber, where they are thoroughly mixed and broken down to a fine hydrate. An auxiliary water spray pipe in the finishing chamber provides the additional water required, and the material leaves the hydrator virtually dry and pure. Foreign matter such as unburnt limestone, slag, coke, clinker, etc., is mostly retained in the screen, and is discharged by chute to a receptacle through a port and chute at the opposite end to the feed mouth. Small unslakable particles in the hydrate can be eliminated by sifting the powder, after it leaves the machine, in an air separator. The steam, carrying fine dust, is drawn by natural draught through the water sprays of a patent condenser, which recovers a large proportion of the dust as milk of lime, and also transmits the steam heat to the slaking water, accelerating the slaking process. Dust recovery represents about 5% of the total hydrate produced.

The rotary-kiln lime plant at Abadan on the Persian Gulf embodies a hydrating machine of this type. Here, automatic table feeders are used, and an air separator removes the nibs and impurities. The pure hydrate is then elevated into a storage bin and withdrawn as required by an extracting conveyor. Conversion of the calcium oxide into hydrated lime is effected by adding about 30% of water to the quicklime. The hydrating process here is controlled by the temperature of the hydrate discharged from the machine. It

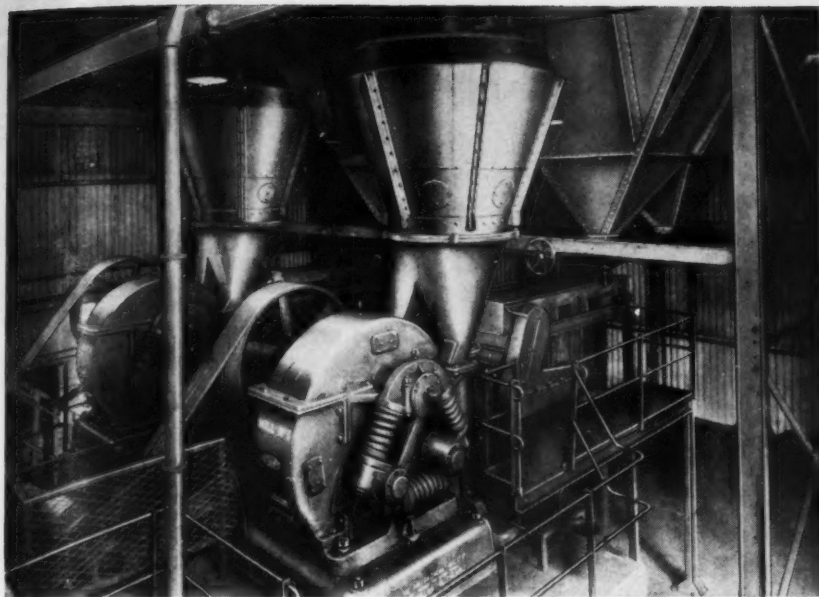
has been found that, if the water supply be adjusted in accordance with the temperature of the hydrate discharged, the percentage of water is about correct. Normally, the temperature is about 100°C.

In contrast to this machine, which is of Swiss origin, but is also made in Great Britain, is an American hydrator of vertical, continuous type, known as the Schaffer. In this, the lime and water fall on to a plate near the top of the hydrator, are stirred up by a number of blades hanging from a horizontal revolving arm and, in the end, pass through an aperture in the plate to a corresponding plate below. Four, six or more of these plates, provided with scrapers, are set one below the other in the machine, ensuring a thorough mixing of the lime and the water, and the mixing period is longer than with the majority of hydrators. Steam and air pass up the chimney. The machine will produce about 18 tons/hr. of high-quality hydrated lime, but, although efficient, it is expensive. As the crushed lime is delivered into the hydrator in a steady stream from an automatic weighing belt, water is automatically added in proportion to the weight of the lime. As long as the lime is of suitable quality, therefore, there cannot, in theory, be too much water added. The stirring rakes also facilitate mixing without bringing the lime into too much contact with water. A second water spray can be added where water is required in two separate amounts, one at the beginning of slaking and the other later.

The rotating plates have rims that retain the weightier, imperfectly slaked grains, while the lighter pass over them without difficulty. This speedily sorts out the



Ball mill with top half of dust casing and one outer sieve removed.



[Courtesy: British 'Rema' Mfg. Co., Ltd.]

Ring roll mill plant arranged on the storage system with totally enclosed automatic dust filter.

particles needing a longer slaking period, and makes it easier to give them the additional treatment they require. A pressure- and temperature-regulating collar saturate the atmosphere in the upper part of the hydrator with steam, and this atmosphere is under slight pressure. The lime, after hydration, is cooled and excess moisture eliminated by the passage of an air current through the machine.

Principles of lime hydration

Certain principles must be borne in mind when employing lime hydrators. First, adequate time must be allowed for the slaking operation to be completed. A lime hydrator does not slake lime, it merely allows lime to be slaked. In other words, it is essentially a container in which a chemical process goes on, not itself an active agent in the process. The use of water is important. Where some limes are concerned, the water is better added in one amount. Others need a quantity of water to develop high temperature first, and more water added later to keep up this temperature. Too much water must never be added at once, so as to avoid unslaked lime, caused by too low a temperature. The amount of water may be affected by the temperature of the hydrator itself which, after a long period of continuous operation, becomes higher than when starting from cold, and therefore needs extra water. It is this fact that tends to make automatic water-adding devices less satisfactory than they should be in theory.

A lime slow to slake at the outset may be 'started' effectively with hot water or steam. Steam pressure in the machine should never be allowed to rise above 110 lb./sq. in.

Screening

When the hydrate has been produced, it is normally conveyed to and kept in bins for a period of at least 48 hr. before being fit for service. However, as stated earlier in connection with the Abadan plant, it is advisable before the hydrated product is used to separate out the nibs and coarse particles. This can be done either through screens or by means of an air separator. If screens are employed, a 50 mesh is generally used, the angle of inclination of the surface of the screen being modified in accordance with the degree of fineness required. In some instances, the hydrate is caused to pass through an automatic pulveriser with air separation and reject. A more up-to-date tendency in the U.S. is to deal with this material by means of air separators in series, the tailings from the final separator being discarded or sold for purely agricultural use. A variation on this system is to feed the tailings to an automatic pulveriser with a reject for final cleaning. Very clean tailings can be had in this way, being cleaner than in an air-separation unit. In Table 3 some figures for a typical air separator are given.

TABLE 3

Overall width ft. in.	Overall length ft. in.	r.p.m.	Screen surface ft.
7 3½	6 11½	250	4 × 6
11 3½	6 11½	250	4 × 6

H.P.: 3 and 4 respectively

Composition of lime hydrate

The formula for lime hydration is, of course, $\text{CaO} + \text{H}_2\text{O} = \text{Ca(OH)}_2$. The lime hydrate corresponds to 32.1% by

weight of the quicklime, and consists of 75.7% lime and 24.3% water. The hydrate has a specific gravity of 2.078. Impurities consist of silica, alumina, iron oxide and fragments of uncombusted lime carbonate, and their presence decreases the expansion of volume consequent upon slaking. If the water is added at one operation, the total expansion will be 3.5 times the original volume, but if the water is added by degrees, the volume expansion will be much less, declining to as little as 1.7 times for slaking in air. The exact expansion is, as stated, dependent on the total percentage of impurities contained in the hydrated product.

Efficiency of processes

One of the great problems is that the effectiveness of a hydration process cannot be determined in advance, being governed by the type of lime used and available. Many manufacturers put down a hydration plant, and assume that all they have to do is to go right ahead at top speed. Whatever the claims made by makers of plant for their apparatus, the efficacy of any particular hydrating system or machine can only be determined after a long period of operation. The product obtained is governed by the volume of material in the machine, the period required for its complete permeation by the water, and the skill and experience of the operator. A plant that is demonstrated to be most efficient in one set of circumstances may prove totally unsuitable for a different set of conditions. Hence, it is always advisable to carry out preliminary tests involving the hydration of at least a ton of the available lime. These should be carried out in the actual machine it is proposed to purchase, and the result judged by the fineness and freedom from unslaked lime of the product, the cost per ton of the finished product (with a suitable allowance included for wear and tear of plant) and the degree of skill, experience and inspection necessary to produce a satisfactory material.

Lime hydration is both a mechanical and a chemical process and, as such, demands a good deal of competent supervision. The more variable in its properties the lime used the greater will have to be the degree of supervision, whereas if the plant employed has been wisely chosen after careful test, and the lime is consistent in its behaviour under slaking conditions, supervision can be less exacting.

Wherever the lime employed is of magnesian or other slow-slaking type, it is advisable to employ silo storage to give adequate time for the completion of the process. No machine can rapidly and thoroughly slake a lime of this type in a brief period. Another point to be borne in mind is that it is unreasonable to demand a guarantee of free moisture percentage, because the determination of such a percentage is virtually impracticable. Claims that products produced by a particular

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A New Dephenolisation Process for Low-Temperature Carbonisation Plants

By W. Lowenstein-Lom

It has been found difficult to obtain an indigenous solvent to purify the ammoniacal liquors, particularly to remove phenols, of low-temperature carbonisation plants. It used to be thought that most light fractions of the oils were unsuitable. However, the author has found that, particularly in the presence of organic bases, certain fractions can be used and are even preferable to benzole and solvents obtained by high-temperature tar distillation.

THE purification of ammoniacal liquors and other effluents from coal carbonisation works has been approached from various angles and offers no difficulties under standard conditions. From the point of view of river pollution the removal of phenols is by far the most important of the various purification processes. The high oxygen absorption of phenolic substances, together with their water solubility and consequent high concentration in effluents renders their removal absolutely essential. A maximum concentration of 0.2 p.p.m. is generally permissible and does not tax the self-cleaning capacity of rivers and streams excessively. Obviously this cannot directly be achieved by commercial phenol recovery methods.

It follows that either extensive dilution of effluents must take place after phenol extraction or alternatively some destructive dephenolisation process must be resorted to. The latter include biological oxidation, e.g. activated sludge processes, coke quenching, evaporation of liquors, using them for coal flotation, etc.^{1,2}

The composition of phenolic extracts from liquors of various origins shows certain differences. In general it can be said that the higher the temperature of carbonisation the smaller the concentration of dihydric and polyhydric phenols. In medium- and high-temperature tars carbolic acid and cresols definitely predominate and already the proportion of xlenols (comprising actually ethyl and propyl phenols as well) is small. Table I shows a comparison of four different phenolic extracts which can be considered typical.

Standard methods

Standard methods for the purification of effluents, the phenolic composition of which corresponds to extracts 1 and 4, are obvious. Benzole, being actually one of the products of high-temperature carbonisation and a good solvent for the lower tar acids, has been used for many years,^{1,2,3,4} and a reduction of phenol contents down to 0.2 g./l. can be achieved by its use. Similarly, various other light fractions of high-temperature coal tar have been used, apart from oils stripped from the gas stream.¹⁰ Effluents and carbonisation liquors from the low-temperature carbonisation of hard lignite, e.g. the Bohemian

TABLE I

Phenolic extract from	1 High temp. bit. coal tar	2 Boghead l.t. oil	3 German lign. l.t. tar	4 Bohemian lign. l.t.
	%	%	%	%
Phenol	18.9	14.2	12.5	13.5
o-Cresol	13.2	9.5	8.0	5.8
m-Cresol	11.4	7.1	7.8	3.9
p-Cresol	12.3	10.7	8.5	4.1
Xlenols	21.3	20.2	18.2	6.3
Pyrocatechol and homologues ..	0.6	1.9	5.6	25.4
Resorcinol and homologues ..	2.1	7.5	8.9	8.3
Higher boiling tar acids ..	15.3	21.7	20.3	22.1
Residue	4.9	7.2	11.2	10.6

TABLE 2

	Fraction 1	Fraction 2	Fraction 3
Hydrocarbon composition:	%	%	%
Aromatics	39.8	43.2	46.8
Naphthenes	27.5	28.4	30.8
Paraffins	16.9	18.2	18.3
Olefines	15.8	12.2	4.1
Distillation curve:			
Under 90°C.	5.2	—	—
100°C.	17.3	—	—
110°C.	30.5	—	—
120°C.	45.1	3.6	—
130°C.	61.8	10.2	—
140°C.	76.7	31.7	—
150°C.	81.5	59.4	5.2
160°C.	86.1	71.6	15.8
170°C.	90.0	80.3	29.2
180°C.	93.9	86.1	43.7
190°C.	97.4	92.4	63.2
200°C.	99.0	97.6	78.4
210°C.	—	98.9	85.3
220°C.	—	—	93.2
230°C.	—	—	98.5
240°C.	—	—	—
Aniline coefficient	1.24	1.30	1.40
Reid vapour pressure	10.7	8.0	6.2
Density	0.802	0.815	0.832

variety, are not amenable to phenol extraction by benzole or similar solvents. The higher phenols, particularly catechol, are much too soluble in water and their distribution coefficient between benzole and water is unfavourable. Consequently, new methods of dephenolisation had to be devised, paramount among these being the tricresylphosphate^{5,9} and phenosolvan processes.^{5,9,7}

A difficulty arises in the case of low-temperature carbonisation of ordinary lignites and bituminous coal, particularly bogheads. There the composition of phenols in tar and effluent corresponds to that of horizontal retort tars. The percentage of catechols is insignificant and

that of resorcinols is small. Benzole and other coal-tar solvents would consequently be preferred extracting agents. But none are actual products of the process and therefore have to be imported into the plant. In view of fiscal and other difficulties this is undesirable.

Similarly, tars belonging to group 4, after partial hydrogenation yield considerable quantities of phenol-containing liquors. In these the more easily reduced polyhydric phenols have been converted into monohydric phenols, aromatics and naphthenes already in the first stage of hydrogenation. The mono-phenols, on the other hand, have undergone no change and sometimes form up to 1% of the liquor in

TABLE 3. FRACTION I

	Concentration in H ₂ O, g./l.	Concentration in solvent, g./l.	Distribution coefficient	Average
Phenol	10.82 1.03 0.095 0.010	27.30 0.927 0.095 0.002	2.5 0.9 1.0 0.2	1.2
<i>o</i> -Cresol	10.05 0.990 0.095 0.010	52.40 4.95 0.380 0.032	5.0 4.0 3.2 5.0	4.3
<i>m</i> -Cresol	9.950 1.020 0.090 0.010	49.75 5.200 0.350 0.030	5.1 3.9 3.0 4.5	4.2
<i>p</i> -Cresol	10.02 1.00 0.105 0.009	45.09 4.50 0.360 0.029	4.5 3.4 3.2 2.1	3.9
Xylenols	10.01 1.010 0.096 0.010	21.00 2.020 0.180 0.018	2.0 1.9 1.8 0.6	1.9
Catechol	5.20 0.50	3.12 0.02	0.4 0.5	0.5
Resorcinol	4.90 0.50	2.45 0.25	0.5 0.5	0.5

TABLE 4. FRACTION 2

	Concentration in H ₂ O, g./l.	Concentration in solvent, g./l.	Distribution coefficient	Average
Phenol	10.82 1.03 0.095 0.010	48.75 3.61 0.294 0.029	4.5 3.5 3.1 2.9	3.5
<i>o</i> -Cresol	10.05 0.99 0.095 0.010	120.8 10.49 0.884 0.089	12.0 10.6 9.3 8.9	10.2
<i>m</i> -Cresol	9.950 1.020 0.090 0.010	83.60 6.33 0.450 0.048	8.4 6.2 5.0 4.8	6.1
<i>p</i> -Cresol	10.02 1.00 0.015 0.009	86.10 6.30 0.565 0.043	8.6 6.3 5.1 4.8	6.2
Xylenols	10.01 1.01 0.096 0.010	149.1 11.01 0.884 0.086	14.9 10.9 9.2 8.6	10.9
Catechol	5.20 0.50	4.67 0.251	0.9 0.5	0.7
Resorcinol	4.90 0.50	4.420 0.351	0.9 0.7	0.8

TABLE 5. FRACTION 3

	Concentration in H ₂ O, g./l.	Concentration in solvent, g./l.	Distribution coefficient	Average
Phenol	10.82 1.03 0.095 0.010	43.40 3.61 0.304 0.028	4.0 3.6 3.2 2.8	3.4
<i>o</i> -Cresol	10.05 0.990 0.095 0.010	110.8 10.39 1.036 0.108	11.0 10.5 10.9 10.8	10.8
<i>m</i> -Cresol	9.95 1.02 0.090 0.010	101.3 8.26 0.585 0.044	10.2 8.1 6.5 4.4	7.3
<i>p</i> -Cresol	10.02 1.00 0.105 0.009	91.20 9.00 0.861 0.065	9.1 9.0 8.2 7.2	8.5
Xylenols	10.01 1.010 0.096 0.010	140.0 11.31 0.970 0.071	14.0 11.2 10.1 7.1	10.6
Catechol	5.20 0.50	5.21 0.302	1.0 0.6	0.8
Resorcinol	4.90 0.50	4.415 0.351	0.9 0.7	0.8

contact with the hydrogenated oil. Again, all such hydrogenation plants find themselves in the predicament of having to use a non-indigenous solvent, benzole or some other light aromatic oil, and it appeared highly desirable to replace the latter by a cheaper dephenolising agent of their own production.

Tests made

A number of intensive tests were made with various straw-oil distillates, the phenols of which had previously been removed by an alkaline wash. In order to define the extracting agents as accurately as possible, type analyses of the oils were carried out and their distillation curves (A.S.T.M.) and other characteristics determined. These are summarised in Table 2.

Solubilities and distribution coefficients between water and the solvent for carbolic acid, *ortho*-, *meta*- and *para*-cresol, a mixture of xylenols, catechol and resorcinol, each for several different concentrations, are arranged in tables 3 to 6. Comparisons with technical benzole and pure benzene in Table 7 show that most of these oils are equal and the fractions numbered 2 and 3 even superior to benzole, particularly with regard to the extraction of carbolic acid and *ortho*-cresol. As the normal solubility of phenols in naphthenes and paraffins is not better than in aromatics, this was somewhat surprising and an explanation was required.

Effect of sulphuric acid wash

It was observed that the distribution coefficients and extracting power were considerably reduced by a previous sulphuric acid wash of the solvent. In order to establish whether this was due to the removal of organic bases or rather to a removal or condensation of unsaturated hydrocarbons, fraction 3 of the above-mentioned solvents was shaken up with 15% sulphuric acid before extraction and subsequently 2.3% of a mixture of pyridine bases were added. In Table 7, distribution coefficients for the three cases are compared, and it is found that the extracting power of the solvent, weakened by the sulphuric acid wash, is practically restored by the addition of the bases. An easy way of improving phenol extraction had thus been found, and in the last line of the table the effect of the addition of a small percentage of bases is shown.

The organic bases are not removed if the oil is used for phenol extraction. Before the extraction phenols are removed from the oil by a caustic wash, and if there is an excess of oil production, the oil is returned after phenol removal, and fresh oil is used for each extraction. Occasionally it was observed that a gradual rise in extracting power of the oil occurred if the oil was recirculated and used for several extractions in succession. It appears that a gradual enrichment from pyridine bases in the ammoniacal liquor takes place and thus extraction conditions of the solvent are improved. Incidentally, the reason for

tration to 10 to 15 mg./l. Further dilution by other plant effluents and river water brought this down to below 1 p.p.m.

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The Hydration of Lime

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appliance provide a certainty of minimum free moisture content, especially if a very low figure is quoted, should be regarded with suspicion.

The fineness of the eventual product depends largely on the country of origin. In Great Britain, it is usual to offer a product of which 95% will pass through a 200 mesh. Somewhat characteristically, the Americans often claim and may even supply where essential 99% passing through a 300 mesh.

The storage of the product in paper bags or sacks can be adopted for indefinite periods as long as completely dry conditions obtain. Standard tests for the material have been drawn up and have been accepted.

One point not hitherto mentioned is the importance of avoiding contact of the lime with the air in the hydration process from beginning to final air separation. Closed-circuit grinding mills are therefore advantageous, as are a suitable hydrator and air separator in which the original volume of air can be used over and over again. Access of air to the material results in undesired carbonation, even to the extent of 8% of calcium carbonate, which may render the lime totally unsatisfactory for certain uses. Similarly, it is important that whatever air is admitted into the plant should be as pure as possible. This means that the hydrating plant must be located as far as possible from any source of pollution of the atmosphere, such as rotary or other kilns, furnace or boiler stacks, rough grinding plant, open fires, etc.

Bromine and Magnesium from the Sea

MAN'S hope to develop power from the sea has not yet been realised, but the prospect of recovery of minerals from that mighty storehouse has long since become real. Recent large commercial projects for recovering elemental bromine and magnesium from sea water in the U.S. have been described by C. M. Shigley of the Dow Chemical Co. in the January 1951 issue of the *Journal of Metals*. The following summary appears by courtesy of *Engineering Newsletter*.

The total volume of the oceans is estimated to be 320,000,000 cu. miles. Although the salinities of the several seas vary somewhat, the average is approximately 35,000 p.p.m. of dissolved salts, equivalent to 165,000,000 short tons per cu. mile. The oceans of the world thus represent a storehouse of about 50 million billion tons of dissolved materials. The chloride ion represents 54.8% of the total salts, the sodium ion 30.4%, sulphate 7.5%, magnesium 3.7%, calcium 1.2%, potassium 1.1%, carbonate 0.3% and bromide 0.2%. Although the sea is believed to contain at least traces of every element, these eight ions account for over 99% of the sea-water salts; all other elements total less than 1%.

Recovery of salt

Since the sodium and chloride ions represent 85% of the dissolved salts and are the most easily extracted, it is not surprising that they have been involved in the first recovery on record. Common salt was undoubtedly the first compound to be removed from sea water and used by man. It is believed that salt was used by cave men at least 5,000 years ago. Salt from sea water is mentioned in Chinese writings about 2200 B.C. Aristotle in his *Meteorologica* wrote of the origin and usefulness of the salts of sea water, and described a method of de-salting sea water. The ancient Greeks, Romans and Egyptians were familiar with production of salt by solar evaporation of sea water. Such salt recovery has been common in China, India and Japan for many centuries and still continues. Salt from sea water was produced on the Atlantic coast of North America about 1680 and on the Pacific coast in 1852. The Atlantic coast industry was short-lived, but that on the Pacific coast has thrived to this time.

Other minerals

The production of crude soda and potash from the ashes of seaweeds was accomplished in Scotland as early as 1720. Iodine was recovered from sea weeds early in the 19th century; magnesia was first prepared on the Mediterranean coast at the end of the century. The records do not indicate any additional progress until

1923, when magnesium chloride and gypsum were produced from the bitterns from solar evaporation of the sea water of San Francisco Bay.

In 1926 the first sea-water bromine was recovered on a small commercial scale by chlorinating the San Francisco Bay bitterns, steam stripping, condensing and purifying the product.

In 1931 the production of potassium chloride by evaporation of the waters of the Dead Sea was inaugurated. In 1932 bromine was recovered on a commercial scale from the residual liquors of the potassium plant, using a process similar to that employed for San Francisco bitterns.

Recovery techniques

Prior to 1933 the survival of the majority of the projects recovering material from sea water depended upon solar evaporation for initial concentration of the valuable sea salts. A few projects depended on adsorption or biochemical concentration, as in the production of soda, potash and iodine from seaweed. Of the other likely methods of recovery, precipitation by a specific reagent had been demonstrated in the commercial production of magnesia by liming sea water, and in the experimental removal of bromine as the insoluble tribromo aniline. The latter process was developed in 1924 by the Ethyl Gasoline Corporation, and carried to large-scale experimental work in the floating chemical laboratory, the S.S. *Ethyl*. Separation by ion exchange processes or by selective volatilisation of the material sought had not yet been commercially exploited.

Bromine extraction

In 1933 a sharp increase in the demand for ethylene dibromide as a constituent of petroleum anti-knock could not be met readily by increasing the output of bromine plants using subterranean brines. Based on experimental work done in anticipation of this need, a plant was constructed by the Ethyl-Dow Chemical Co. at Kure Beach, North Carolina, to remove bromine directly from sea water, without the prior concentration which had been necessary for the earlier commercial recoveries. The significant feature of this operation was that the small amount of bromine was removed as a gas from the relatively large volume of water. Past efforts had been directed largely towards removal of large amounts of water as vapour from the relatively small amounts of dissolved salts.

The original Kure Beach plant was designed to extract 6,000,000 lb. of bromine p.a. for the production of ethylene dibromide. Through minor additions and process improvements, the capacity was increased to nearly 9,000,000 lb. p.a. In 1937 the capacity of the plant was doubled

and, in 1938, increased again, reaching an output of approximately 40,000,000 lb. p.a.

In 1940 a further increase in bromine requirements led to the erection of a plant at Freeport, Texas, to recover bromine from the waters of the Gulf of Mexico. This plant had an initial capacity of about 30,000,000 lb. of bromine p.a., and a second unit of equal output was built in 1943.

Magnesium

Another milestone in the recovery of minerals from sea water was passed in 1941, when at Freeport, Texas, the first magnesium metal was produced from water of the Gulf of Mexico by the Dow Chemical Co. Although the precipitation of magnesium hydroxide from sea-water bitters and brines and the method of making magnesium metal from magnesium chloride were both well known prior to 1941, it was not until then that these methods were revised and integrated to give an economically feasible process for making metallic magnesium from sea water. The success of the first 18,000,000-lb. p.a. magnesium plant led to the erection of another plant of equal size one year later.

In 1942 a 72,000,000-lb. p.a. magnesium metal-from-sea-water plant was built by the U.S. Government at Velasco, Texas, as a part of the programme planned to meet emergency war-time needs. The plant was designed and run by the Dow Magnesium Corporation. It operated at or above rated capacity for the duration of the war. That sea water represented no handicap as a source of raw material for the newly developed magnesium process was demonstrated by comparative costs published by the Defence Plant Corporation after cessation of hostilities. The Velasco plant bettered by nearly 30% the lowest cost achieved by other Government plants using more concentrated magnesium sources.

Unfortunately, both the Government-owned magnesium project at Velasco and the privately-owned bromine plant at Kure Beach were among the war casualties when war-time production capacity encountered reduced peace-time demands. However, economic survival was largely in favour of the sea-water processes. Since return to peace-time conditions, the entire U.S. production of virgin magnesium and an estimated 80% of the bromine have been derived from sea water.

Plant siting

The processes successfully used for bromine production at Kure Beach and Freeport, and for magnesium at Freeport and Velasco are chemically very simple. They have been described in detail in articles by Stewart, Kirkpatrick, Schambra and others.

The proper location of a plant utilising either bromine process is important to the success of the project. A place is required where sea water of high and constant salinity is conveniently available, free from

organic contamination, and undiluted by major fresh-water rivers. It also must possess favourable circumstances for disposing of the large quantities of processed water without mixing with the unprocessed water. Where shallow water and variable currents prevail, the intake and effluent systems should be widely separated. Deep water alongshore and constantly favourable shore currents lessen the need for such separation. A plant site slightly above sea level is preferable to reduce pumping costs.

Since both processes depend on vapourisation of bromine and since the vapour pressure of bromine in sea water varies considerably with temperature, a location in a warm climate is desirable. Other things being equal, a blowing-out tower handling 25°C. sea water can operate at a higher rate and can produce approximately twice the amount of bromine as the same tower operating at 10°C. The absorption of the alkali process also is susceptible to temperature effects. Absorber losses are 5 to 15-fold more at 10°C. than at 25°C., depending on the excess alkalinity of the absorbing solution. A location as near as possible to the source of economical raw materials and power, and to the point of disposal of the finished product, is desirable, but other factors are of secondary importance when compared with the need for favourable oceanographic and climatic conditions.

Operational control

Because of the relatively large quantities of raw materials that must be handled to obtain each pound of bromine, it is necessary that close operational control be maintained at all points. Since reliable indicators and automatic controls have made a large contribution to the success of the large-scale recovery of bromine from sea water, they must be regarded as integral parts of both processes.

The bromine process known as the 'acid process' or SO_2 process was developed in 1937, and has been used in all bromine-from-sea-water plants built in the U.S. since that date.

The manufacture of magnesium from sea water is quite different from bromine processes. Magnesium is taken out of the sea water in an alkaline condition instead of acid, and is removed by precipitation rather than blowing out.

Economic aspects of magnesium extraction

The economic factors involved in the magnesium-from-sea-water operation are somewhat different than those of bromine. From an oceanographic or climatic standpoint, location is not as critical. The sea water that must be processed per lb. of magnesium is only 5% that required per lb. of bromine, and water temperature has little effect on the magnesium recovery. More important is a location favourable to the supply of raw materials and power. The convenient availability of lime, and

abundant and inexpensive fuel and power, obviously are essential for competitive operation.

The process can achieve a recovery of 85 to 90% of the magnesium in sea water. The performance of each step represents a compromise between high efficiency and high capital cost, and the justifiable recovery must be calculated for the conditions of each plant. The process has the inherent advantage that the majority of the materials can be conveyed by pumping. Most steps are continuous and subject to the benefits of automatic control.

In conclusion, the large-scale recoveries of bromine and magnesium from sea water must not be regarded as merely incidents in the record of scientific progress of the past three decades. They are indicative of a pronounced trend towards using the seas for more of life's needs.

New standards

Graphical symbols. The system of instrument symbols described in a new British Standard provides a means of graphical representation suitable for use in any engineering or process-line diagram. It is also suitable for use in engineering and industrial reports and in plant arrangement and layout drawings.

The system has been devised in simple form to enable any combination of instruments or control equipment to be shown in relation to the units of the plant with which they are associated. It is designed to be flexible so that alterations of instrumentation at successive stages of design may be readily shown on a line diagram in such detail as the system permits. Three diagrams are included only to illustrate the application of this system of symbols.

The notation used with the symbols provides a general indication of the kinds of instruments and controls represented. These indications should be supplemented by more detailed and informative data sheets or 'instrument lists,' covering the exact specifications of the equipment required.

Copies of Graphical symbols for instrumentation (B.S. 1646:1950) may be obtained from the British Standards Institution, 24 Victoria Street, London, S.W.1, price 2s. 6d., post free.

Pipes for pressure vessels. Following the publication of B.S. 1500, 'Fusion-welded pressure vessels' (see INTERNATIONAL CHEMICAL ENGINEERING, November 1950, pp. 519-521), the British Standards Institution has just published in one document two standards, B.S. 1507 and B.S. 1508, 'Ferrous pipes and tubes for pressure vessels for use in the chemical and allied industries.' It contains comprehensive specifications for pipes and tubes manufactured from 12 types of steel covering mild steel, alloy steel and austenitic steel. The standard costs 10s. 6d. from the Institution, as above.

VACUUM TECHNIQUE

Sources of gas in vacuum systems and methods of control

By S. L. Martin, M.Sc., F.R.I.C.

IN any vacuum process a first problem of design is the selection of a suitable pumping system which allows the desired degree of vacuum to be attained in an economical time. For this a certain quantity of air must first be removed in a definite time, the absolute quantity depending on the volume to be evacuated and the ultimate pressure desired. Sufficient data is available on speeds of various pumps at different pressures and on the resistance of pipes and tubes in relation to their diameter and shape for accurate correlations of evacuation time and desired speed of pumps to be made in this respect. However, there still remains the problem of coping with gases or vapours which were not originally in the space of the system, but which enter this space—or might enter it—from one or more of the sources which will be indicated below. The extent of this problem depends largely on the nature of the process and on the ultimate degree of vacuum desired. Obviously, in a continuously pumped or dynamic system the problem will exist only until the rate at which the pumps remove gas from the space equals the rate at which gas enters it at the desired pressure, for thereafter this pressure will be maintained. On the other hand, in a static system not continuously pumped, pumping must continue until the rate of entry of gas into the system when shut off from the pumps is low enough for the desired pressure not to be exceeded during the time the process is in operation. It follows also that the higher the allowable pressure the higher will be the permissible rate of entry for either type of system, always—even in a dynamic system—provided that the products of the process are unaffected by the entering gases.

Thus this second problem may be a relative one, serious to the nuclear physicist desiring an evacuated space practically free of molecules other than those he chooses to put in it, and comparatively simple to the engineer running a vacuum evaporation or dehydration process. But that it is a real one is evidenced by the fact that even with an all-glass laboratory system newly set up and free of leaks from the air, it may take days to obtain a 'sticky' vacuum on a McLeod gauge, that is, better than 10^{-5} mm. Hg. Obviously,

major leaks in the system will prevent the attainment of high degrees of vacua, but, excluding their occurrence, this entry of gases into the vacuum space through minor leaks and from other sources is often responsible for the major part of the evacuation time.

The sources of gases and vapours in vacuum systems may be broadly classified as follows:

- (1) Materials, both those of construction and those involved in the process itself. These may:
 - (a) Have appreciable vapour pressures at the operating temperature, and
 - (b) Contain adsorbed or dissolved gas.
- (2) Leaks from the outside air, either:
 - (a) Through the main constructional material or
 - (b) More likely through joints, seals, valves, doors, etc.

The expanding applications of vacuum technique demand larger and larger vacuum vessels, volumes greater than 100 cu. ft. not now being uncommon, and more facilities for external control of processes requiring transmission of mechanical power through the walls of the vacuum vessel. This demand has stimulated the design of leak-proof welded joints, shaft seals, valves, flanges and portholes,

the search for efficient gasketing and sealing materials and the development of satisfactory methods for testing all types of joints for leaks *in situ*. Attention is also being given to the general vacuum properties of new materials, that is, their rates of evaporation and of gas evolution under vacuum at different temperatures.

Gas or vapour from materials of construction

For the main body of vacuum vessels, pumps, gauges and pipelines the chief materials used are glass and steels. However, many other materials find special uses which may be classed as constructional—ceramics for furnaces or as insulators; metals such as copper, nickel, tungsten, molybdenum and iron-based alloys for electrical leads; mica and polymers such as polystyrene for electrical insulators; lead and tin in soldering compositions; mercury and organic oils for diffusion pumps and manometers; greases and oils as lubricants for taps and valves; soft metals such as lead, and rubbers, natural and synthetic, for gaskets; and materials such as phosphorus pentoxide for desiccants. All these may be potential sources of gas or vapour, depending on their nature, past history and temperature of operation.

Vapour pressure of materials

Any substance will boil, that is evaporate freely, when its vapour pressure equals the pressure of the surrounding atmosphere. Therefore, where a vacuum of better than 10^{-5} mm. Hg, for example, is desired, no substance with vapour pressure at its operation temperature much in excess of this should be presented in the vacuum system. The vapour pressure of a substance can to a first approximation be expressed as a function of temperature

TABLE I. VAPOUR PRESSURES OF INORGANIC MATERIALS (BASED ON DATA GIVEN BY DUSHMAN.¹ VAPOUR PRESSURES (V.P.) IN MM. HG)

Material	M. pt. Tm °C.	V.P. at Tm in mm. × 10 ³	Temperatures in °C. where v.p. equals		V.P. at 20 °C.
			10 ⁻¹ mm.	10 ⁻⁵ mm.	
Calcium	810	875	700	408	
Lead	328	5×10^{-3}	832	480	
Zinc	419	160	405	211	
Tin	232	0	1,373	823	
Aluminium ..	660	$\sim 10^{-4}$	$\sim 1,200$	~ 780	
Copper	1,083	0.3	1,432	946	
Nickel	1,455	4.4	1,679	1,157	
Iron	1,535	37.2	1,602	1,049	
Molybdenum ..	2,622	22		1,923	
Tungsten	3,382	17.5		2,554	
Mercury	-38.9	2.5×10^{-3}	82	-24	$\sim 10^{-3}$
Water (ice) ..	0	4,580	-39	-108	17.5
Solid CO ₂ ..	-78.5	760,000	-147.5	-180.5	
Phosphorus pentoxide ..	358 (sub.)		149	42	$\sim 5 \times 10^{-7}$
Alumina	2,050	347			
Silver chloride ..	455		790	479	

(T in °K.) by an equation of the form:

$$\log_{10} p = A - B/T$$

where A and B are constants for any substance, dependent only on the units of p .

From such a relation determined at higher temperatures where the vapour pressures are more easily measurable, the temperatures at which the vapour pressures are of specified orders, such as 10^{-5} or 10^{-1} mm. Hg, can be calculated. For most inorganic materials with which we are concerned, A has a value of 14 to 16, p being measured in mm. Hg, and B one of 15,000 to 30,000, corresponding to negligible vapour pressures at ordinary temperatures. Table 1, compiled from data given by Dushman,¹ shows the approximate temperatures at which p is 10^{-1} and 10^{-5} mm. Hg, the boiling point and p at the boiling point for selected materials, and the vapour pressure at room temperature for more volatile materials.

It is worth remembering that the rate of evaporation, m in g. cm.⁻² sec.⁻¹, of a substance of molecular weight M g. at a temperature T °K where its vapour pressure is p mm. Hg, can be obtained from the relation:

$$m = 0.0585 \times (pM/T)$$

The data on organic materials of possible use as pump oils, greases, sealing compounds and gasketing materials, is not as complete or reliable as could be desired. This is primarily because of the difficulty in making measurements at low pressures, and the uncertainty in extrapolating data obtained at higher temperatures and pressures to temperatures of 20 to 100°C., more particularly with these materials which may have significant vapour pressures at these lower temperatures. In the few cases where the vacuum properties of sealing and gasketing materials have been examined,²⁻⁵ empirical methods were used giving relative data on total pressures from gas or other impurities as well as any pressure from the vapour of the material itself; the results will therefore be discussed in the next section.

Desiccants and freezing traps

If desiccants are used to remove water vapour without the aid of a freezing trap, it must be remembered that these leave a positive vapour pressure of water in the system. At 25°C. the residual aqueous vapour, using phosphorus pentoxide, magnesium perchlorate, potassium hydroxide or calcium chloride, will be respectively 0.002, 0.5, 2 and 140–250 micro g./l. The most frequently used freezing baths are solid carbon dioxide and liquid air, giving temperatures of -78.6 and -190°C. respectively, but as the nitrogen evaporates from the liquid air the temperature rises to the boiling point of liquid oxygen, -183°C. Thus, if air is pumped through a trap immersed in fresh liquid air, condensation may occur and the pumping time will then be lengthened. It is also important to realise that water or other

TABLE 2. DATA ON OILS, GREASES, SEALING COMPOUNDS AND GASKETING MATERIALS (REFERENCE NUMBERS TO SOURCE OF DATA ARE GIVEN IN SUPERIOR FIGURES)

Use	Trade name	Nature of substance	B. pt. at 0.1 mm. (b) or M. pt. (m) °C.	Approx. V.P. at room temp. in 10^{-4} mm. Hg
Rotary pumps	Transformer oil ¹	Petroleum oil		<100
Diffusion pump oils ¹	Apiezon A	" "	144 b	2
	Apiezon B	" "	162 b	0.4
	Apiezon 171 dist.	" "	171 b	0.09
	Amoil S	<i>n</i> -amyl sebacate	146 b	1
	Octoil	Di-2-ethyl hexyl phthalate	150 b	0.3
	Octoil S	Di-2-ethyl hexyl sebacate	174 b	0.01
	Silicones		~200 b	~0.01
Greases	Vacuseal heavy ²	Petroleum base	60 m	10
	Celvacene light ²	" "	90 m	1
	Celvacene heavy ²	" "	130 m	<1
	Apiezon L ¹	" "	<50 m	(~ 10^{-3})
	Apiezon M ¹	" "	<50 m	(~ 10^{-4})
	Silicone ³	Silicones	215 m	<10
Sealing compounds	Apiezon Q ⁴	Carbon and paraffin waxes	<30	100
	Apiezon W ⁴	Pitchlike	60 s	<0.01
	Picein ⁶	" "	80, 105 m	0.01
	deKhotinsky cement ²	Shellac and pitch	50 s	1,000
	Vinyl acetate ⁵	" "		(~0.5)
	Buna S rubber ⁵	" "		(1,000)
	Silastic ⁷	Silicone rubber		(~20)

vapours condensed in traps may give rise to 'virtual leaks' if the level of the freezing agent is allowed to fall very much; the solid condensed out in the upper parts of the trap may then be warmed up to temperatures where they exert significant vapour pressures. Another point is that the vapour may form an aerosol on entering the trap and an appreciable amount may then pass through without being condensed. For this reason it is always preferable to employ a series of traps.

Gas evolution from materials^{8,9}

All forms of glass and all metals will evolve gas on being heated in a vacuum, but the nature and quantities of gas and the temperature of evolution depend on the particular substance and its previous history. With glasses, the gases evolved consist largely of carbon dioxide and water vapour. With metals, hydrogen, carbon monoxide and carbon dioxide are mainly obtained. In all cases the origin is in the manufacturing process and in the adsorption of water vapour, carbon dioxide and oxygen from the air afterwards. We can, therefore, speak of dissolved gas, held within the bulk of the substance, and adsorbed gas, held on its surface. It is quite often difficult to distinguish between the two, but for present purposes this is not necessary.

The carbon dioxide in glasses originates from the carbonates used in their manufacture, and appears to be less the lower the alkali content of the glass and the higher the temperature of fusion. The total quantities of all gases evolved per 100 g. on fusing glasses varies from 10 to 70 c.c. (N.T.P.) with the hard borosilicate glasses such as Pyrex and Hysil, and from 40 to 140 c.c. with the soft soda-lime and lead glasses. With soft glasses the bulk of the gas can be driven off by heating to

about 250°C., but with hard glasses, temperatures of about 500°C. are required; in these two cases the total quantities of gas obtained are of the order of 0.2 to 3 c.c. per 1,000 cm.² of glass surface, and are thus capable of giving a pressure of 0.015 to 0.2 mm. Hg in a volume of 10 l.

Certain metals may hold hydrogen and oxygen in solution. Most metals contain traces of carbon and have a layer of adsorbed oxygen on their surface. If the carbon diffuses to the surface, carbon monoxide and/or dioxide will be evolved on heating. In general, the quantities of potential gas-producing impurities dissolved in the metals of concern here are too small to cause serious trouble at normal temperatures, with the possible exception of hydrogen; however, they may do so if the metals have to be heated to high temperatures in the particular vacuum process. The total volume of gas which can be evolved on heating to high temperatures (above 1,200°C.) per 100 c.c. of any common metal such as copper, nickel, aluminium, iron and steels varies from 4 to 90 c.c. (N.T.P.), 50 to 90% being hydrogen.

The quantity of gas S , in c.c. (N.T.P.) per 100 g. of metal, held in true solution by metals is related to the partial pressure of the gas above the metal, p in mm. Hg, and the temperature T in °K by an expression of the form:

$$\log_e S = A + \frac{1}{2} \log_e p - B/T$$

where A and B are constants for the particular gas-metal pair. Thus S increases directly as the square root of p at constant temperature, and increases exponentially with increasing temperature at constant pressure. There is evidence that oxygen dissolves in copper, iron and nickel, and nitrogen in iron, but in these cases the value of B —which is related to the heat

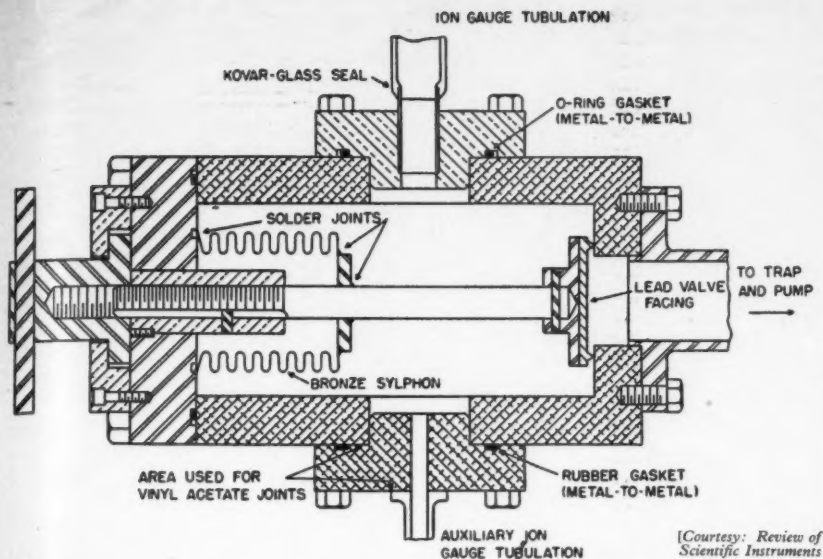


Fig. 1. Sectional view of vacuum chamber illustrating different types of gasketed flanges.

of solution—is such that little or no desorption occurs at temperatures below 700°C. In the case of hydrogen, evolution of gas in a vacuum may commence at temperatures as low as 200°C., and will continue until the amount remaining in solution is equal to that which can be dissolved at the particular temperature and partial pressure of hydrogen in the system. If the system is shut off from the pumps, gas comes off until the equilibrium pressure at the temperature is reached; on evacuating and again shutting off from the pumps, more gas will come off to a slightly lower equilibrium pressure. At low temperatures, this process is slow, being limited by the rate of diffusion of the gas particles within the metal to its surface where they are desorbed. Much of the gas in commercial metals is held in 'blow holes' within the metal, and not in true solution, as evidenced by the sudden rise in gas evolution at the melting points of the metals and the high values for total gas desorbed compared with those for the solubilities of the particular gas. This further lengthens the time for equilibrium to be reached. The rate of diffusion increases exponentially with temperature. Therefore, where metal parts have to be heated in a vacuum process, they should be degassed at temperatures as far in excess of the operating temperature as is possible. Since most metals adsorb grease and oil vapours strongly and since at higher temperatures reaction may occur with carbide formation and hydrogen evolution, it is also good practice to degrease all metal parts within the vacuum space.

Gas from organic materials

Most commercial organic materials contain as impurities traces of solvents or other substances with lower boiling points, that is, higher vapour pressure, than the

main constituent. Furthermore, organic liquids and greases are capable of picking up appreciable amounts of water vapour and gases from the air. The difficulty of obtaining transformer oils with suitably high electrical resistance illustrates this for water vapour, and Dushman¹ quotes for the solubilities of nitrogen, oxygen and carbon dioxide in transformer oil values equivalent respectively to 0.0085, 0.0156 and 0.099 c.c./l. at 1 atm. pressure and 25°C. He also states that for a sample of oil the temperature at which the 'vapour pressure' was 10^{-3} mm. Hg was 34°C. after preliminary heating to remove dissolved air and water, 49°C. after distilling out one-third of the oil, and 77°C. after distilling out about three-fourths. These figures serve to illustrate the likely extent to which water vapour, dissolved gases and volatile impurities may be present in oils not specially treated. These three groups of substances are responsible for the 'total gas content' of organic materials, although it is sometimes difficult to separate their effects from that due to the vapour pressures of the materials themselves—as was pointed out earlier.

All oils to be used in vacuum systems should therefore be first 'conditioned' by vacuum degassing and, if not already done by the manufacturer, fractional vacuum distillation, and should thereafter be kept out of direct contact with the air as far as is possible. The oils used in diffusion pumps—hydrocarbons, esters or silicones—all have a tendency to 'crack' at high temperatures, particularly on metal surfaces, as a result of which gases and/or lower-boiling fractions may be produced during use and may lower the ultimate vacuum attainable unless the oil is reconditioned. They also tend to show some instability to air at the pump-operating temperatures, which makes it undesirable to pass large quantities of air through the

hot pump either in the original evacuation or in letting down the system to air. There is still controversy regarding the relative extents of these tendencies with the three classes of oils, though the silicones appear to have the edge on the others.

Vacuum greases and waxes also contain appreciable amounts of dissolved gases and volatile impurities. It is good policy to keep a grease pot attached to the vacuum system, to melt the grease periodically while pumping, and to leave it in contact with the air for the minimum possible time, since appreciable amounts of gas are taken up by well-degassed grease in a few days. Dissolved gas is generally released by the action of frictional forces on the grease, for example the turning of a tap. The silicone high-vacuum greases, while having other definite advantages such as low vapour pressure at up to 200°C., are difficult to remove from the system during cleaning, and also have lower lubricating properties, in that a fewer number of turns can be made before a tap seizes. Harvey¹³ reports that the low-boiling solvent 1:1 dichlorethane dissolves 20 wt. % of silicone grease at 29°C., whereas tetrachlorethylene, carbon tetrachloride and decahydronaphthalene (*Decalin*) dissolve only 12, 8 and 2% respectively. The addition of about 18% of very fine silica to silicones and other greases has been recommended for improving the lubricating properties.¹⁴

The above general considerations apply also to the solids used as dielectrics or gaskets, but relatively little data is available on their 'gassing' properties as distinct from their vapour pressures or tendency to give leaky joints. Hogg and Duckworth³ examined the rate of rise in pressure in a vacuum system with an ionisation gauge as a function of time for some 30 materials at room temperature. The readings after 70 and 120 min. were expressed as ratios of the readings in a 'blank' experiment, and taken as indications of the vapour pressures, though they undoubtedly included effects due to dissolved gases and lower-boiling impurities. The blank corresponded to a rise in pressure to 10^{-6} mm. Hg after 120 min. The ratios were less than 1.5 for polyethylene, polystyrene, polythene, *Teflon*, acetate cellulose butyrate, *Kodapek* and *Mycalox*; 2 to 3 for *Apiezon W*, *Picein* 105 and acetate cellulose; and greater than 20 for nylon, *Lamicon*, *Bakelites* and glyptal. It was pointed out that a second run after pumping off the gases and vapours would give lower results, as is to be expected.

Dawton¹ considers that all synthetic rubbers have higher gassing rates than natural rubber, but that with continuously pumped systems the rates are not a serious source of trouble after a few hours' pumping. Cloud and Philp⁵ obtained evidence that Buna S rubber in a gasketed flange evolved vapours (which could be condensed out with liquid air and were so distinguished from air leaks) in considerable

amounts. Even after two or three days' continual pumping, the best pressure that could be obtained while pumping was 10^{-6} mm. Hg (no liquid air), an apparent vapour pressure of 10^{-3} mm. Hg at 20°C . being indicated. *Teflon* gave no indication of vapour evolution after 30 hr. pumping, but vinyl acetate continued to give off vapour for over 100 hr., after which a vapour pressure of 5×10^{-7} mm. Hg at room temperature was indicated (10^{-6} and 10^{-4} mm. Hg at 45 and 100°C . respectively). Somewhat similar results have been reported for *Silastic* rubbers Nos. 122, 123, 125, 126, 150 and 160 (Dow-Corning); a vapour pressure at room temperature of 2×10^{-5} mm. Hg was indicated after prolonged evacuation, falling after 30 hr. degassing at 100°C . to about 10^{-7} mm. Hg.⁷ This suggests some advantage for *Silastic*, since it can be safely heated to higher temperatures than natural or synthetic rubbers, but the same authors indicate that if pressures better than 10^{-6} mm. Hg are required, the material is permeable and so unsuitable for use as a jointing material, unless special precautions are taken.

At this point only conclusions regarding the use of these organic solids *inside* the vacuum system are to be made; since still other factors influence their suitability as sealing and gasketing materials, these aspects will be treated in a later section. All the materials require some preliminary degassing when present inside the system, preferably at a slightly elevated temperature. For continuously pumped systems requiring a vacuum no better than 10^{-6} mm. Hg, materials such as *Teflon*, *Silastic*, polystyrene and natural and synthetic rubbers will probably give little trouble, provided they are not positioned where they may become heated during the process; for similar but static systems, a few days' pumping should suffice. For systems requiring higher degrees of vacuum, *Silastic*, polyvinyl acetate and glyptals appear preferable, providing they are first degassed at about 100°C .

Adsorbed gas

All solid substances are capable of adsorbing gases and vapours to some extent on to their surfaces. The strength of the bonding varies considerably for various gas-solid pairs, but in general the quantity of gas or vapour adsorbed varies directly with some power, generally less than 1, of the partial pressure at constant temperature, and decreases exponentially with increasing temperature at constant pressure. At normal temperatures, for all surfaces which are generally considered plain, the evidence points to adsorption to form a mono-layer either of atoms in the case of the elementary gases on metals or of molecules in other cases. Maximum adsorption would then correspond to complete coverage of the available surface with a layer 1 atom or molecule thick. There is also evidence that multimolecular adsorption can occur, particularly with solids

possessing fine pores and with vapours. Thus all metals, with the possible exception of the noble metals, after exposure to air will have a monatomic layer of oxygen adsorbed on the surface. Similarly, all solids will have some water vapour, carbon dioxide and/or oil vapours adsorbed on their surfaces after exposure to normal atmospheres.

The specific surface of a solid, that is its total surface available for adsorption including cracks and pores, may be many times its macroscopic surface. Specific surface areas have been calculated from adsorption data, assuming monolayer formation at first saturation and values for the areas occupied by the gas particles derived from the density of the solidified or liquefied gas. The following figures, quoted by Brunauer,⁸ illustrate the range of values for different materials: Specific surface area of quartz, glass wool, 200-mesh potassium chloride, porous glass and activated charcoal, 0.037, 0.594, 0.24, 125.2 and 775 sq.m./g. respectively; ratios of true to macroscopic surface area for newly polished or bright metals, etched metals, platinum black and evaporated metal (nickel) films, 1.2 to 7, 10 to 70, 1,830 and greater than 2,000 respectively. The amounts of gas required to form a monolayer are of the order of 0.3 c.c. (N.T.P.) per square metre of surface for all the common gases and vapours (calculated from the solid densities), a quantity capable of producing a pressure of about 2×10^{-2} mm. Hg in a volume of 10 l.

These figures illustrate why adsorbed gas must be considered as a problem with metals and porous materials such as ceramics. Similar figures for glass are not available. However, from measurements made at 25°C ., it is certain that with water vapour, even at partial pressures as low as 0.1 mm. Hg, a layer more than 1 molecule thick can be adsorbed by plane glass surfaces after thorough cleaning and vacuum degassing at 400°C . There is a deal of evidence that water vapour may be adsorbed to a total of 3 or 4 molecules thick, carbon dioxide and non-condensable gases to about 1 molecule thick. This truly adsorbed gas can usually be removed rapidly by baking at 200 to 400°C . for short periods, although it appears that further quantities of water vapour and carbon dioxide (originating through partial decomposition of the glass) may be obtained by baking. (The glass may contain traces of hydroxides or hydrated salts, carbonates, nitrates or sulphates, arising from the materials used in manufacture.) Glass is also subject to surface 'weathering' in the atmosphere, the hard borosilicates being better than the soft soda-lime glasses in this, as in other, respects.

The quantity of gas or vapour adsorbed at any temperature is a definite function of the partial pressure. This function has one of the five main forms shown in a paper by Brunauer *et al.*¹⁰ Type I, the Langmuir isotherm, is typical for oxygen on charcoal at

low pressures and for hydrogen, oxygen and most gases on compact metals at pressures well below p_0 , which is the vapour pressure of the liquefied gas at the particular temperature. Types II to V are more often found with adsorption on porous materials such as nitrogen on supported catalysts, bromine on silica gel, benzene on ferric oxide, and water vapour on charcoal respectively. The volume adsorbed at the flat portion in Type I or at the first hump in Types II and IV can be related to the volume required to form a monolayer. For the majority of gas-solid systems of present interest, the isotherms at normal temperatures rise fairly sharply and close to the volume-axis. For instance, at room temperatures, monolayers of water vapour on glass and of oxygen on many metals are formed at equilibrium pressures of about 0.04 and 10^{-5} mm. Hg respectively. In certain cases, particularly with oxygen and carbon dioxide on metals, the gas is strongly held by chemical forces, and cannot be desorbed at room temperature simply by lowering the pressure, that is there is a hysteresis effect with the desorption isotherm; higher temperatures are then required for desorption. In other cases where the molecules are held by physical forces, as with water vapour on most surfaces, desorption will occur to some extent on lowering the pressure below the equilibrium value, but at a slow rate. Thus, if a system is evacuated and then shut off from the pumps, water vapour will come off until the equilibrium pressure is set up in the system. Where thicker layers of water are present, it must be remembered that, due to the loss of latent heat of evaporation from the outer layers, the lower ones will be cooled and will, therefore, evaporate more slowly. The system should be baked out at the highest possible temperature while being pumped, to achieve a good shut-down vacuum in a short pumping time. A difficulty with glass systems in this respect is met in the impracticability of baking out the barrels of taps or the insides of ground joints where greases or waxes are used. This can be lessened by previously baking the taps and joints in air at about 500°C ., before assembly, when the bulk of the adsorbed water vapour will be removed. The baking out of assembled apparatus is usually carried out by torching or by electrically heating ribbons of high-resistance alloys wound around the glass tubing. Stewart¹⁰ has recently suggested painting the glass parts with carbon-black suspensions, covering all taps and greased parts with aluminium foil, and heating up the coated glass parts with infra-red rays from lamps about 1 cm. away from the parts; a temperature of 400°C . can be reached in 5 min., and 1 to 2 hr. is necessary for degassing to a point where an ionisation gauge gives the same reading with lamps on and off.

Because of the rapidity with which the degassed metal and glass parts adsorb

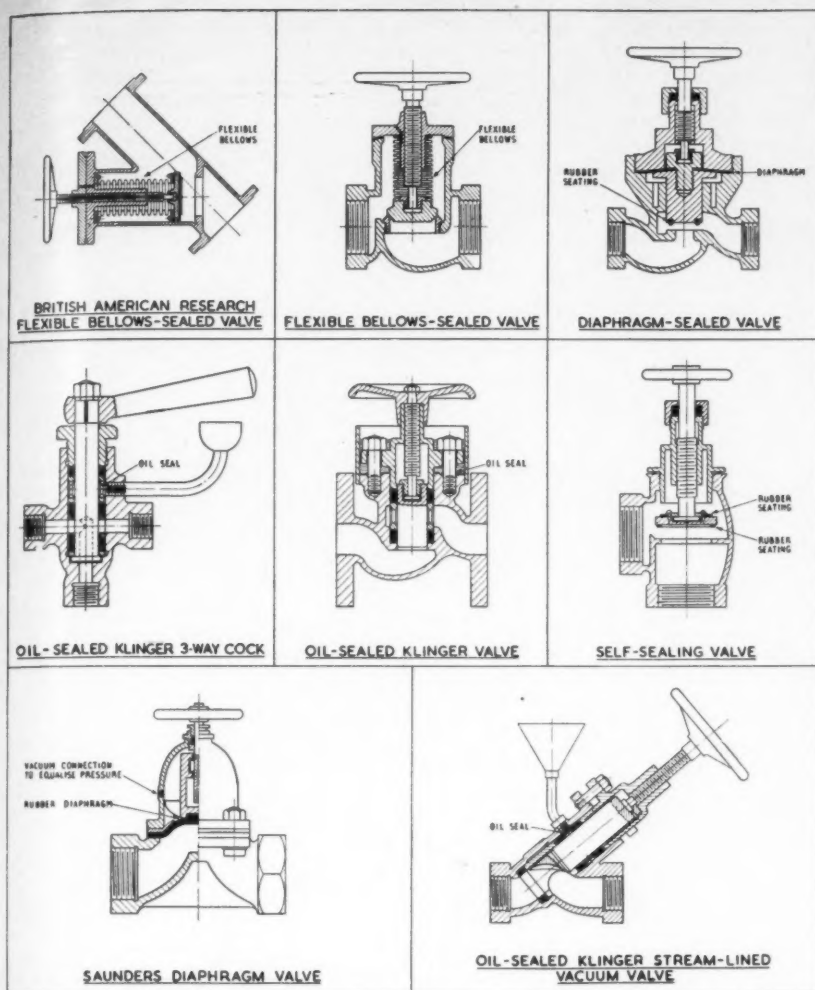


Fig. 2. Examples of some types of vacuum valves.

water vapour particularly, precautions should be taken to admit into the system only dry air, and preferably CO_2 -free air, and the system or parts of it should not be left unduly long in contact with the ordinary atmosphere. Otherwise re-evacuation will again be lengthy. For this reason, too, liquid air and other vapour traps should not be allowed to warm up while opened to the system. It should be possible to shut the system off from them and to evacuate and bake out the traps before opening the system to the pumps. Norrish and Russell¹² have suggested coating the inside of glass parts with a monolayer of a hydrophobic substance, itself stable under vacuum, to minimise adsorption of water vapour. They recommend a 0.05% solution of *Cetavlon*, $\text{C}_{16}\text{H}_{33}\text{N}^+\text{Me}_3\text{Br}^-$, the excess being removed by washing with water. Reference has also been made² to the use of glyptal and silicone varnishes as surface coatings for glass and metal surfaces, followed by a baking-on process to reduce gas adsorption.

Gas entering by leakage from the air

It was earlier pointed out that this might occur because of diffusion through the materials of construction, which would then be permeable to the gas, or because of leaks at permanent or temporary seals.

Permeability of materials⁹

In order for gases to permeate through non-porous materials, they must first be adsorbed at the outer or high-pressure side, must then diffuse through the lattice of the solid, and finally must be desorbed at the inner or low-pressure side. In assessing permeability, care must be taken to eliminate the possibility of leaks through any seals which have to be made in setting up diaphragms of the test materials. Reliable data are available for glasses, metals, ceramics and some rubbers, where special efforts have been made to ensure good bonding, but not on many of the materials used as gaskets, where the empirical methods used have been often incapable of distinguishing between permeation through the material and leakage

at the gasket/metal or gasket/glass junction.

For constant pressure on the outside (partial pressures of oxygen, nitrogen, carbon dioxide, rare gases and water vapour may all be taken as constant for the present purposes) and negligible pressure on the inside, the permeability P , expressed in c.c. (N.T.P.) of gas flowing per second through 1 cm^2 surface of material 1 mm. thick at 1 mm. Hg pressure difference across the material, increases exponentially with temperature. With glasses, helium has the highest permeability of the elementary gases and, other things being equal, its permeability is highest with the hard silica glass and lowest with the soft lead glasses. The value of P for helium/silica at 300°C . is 3.15×10^{-10} c.c. $\text{sec}^{-1} \text{cm}^{-2} \text{mm}^{-1}$ (mm. Hg) $^{-1}$. It can therefore be taken that the inlet of gas by diffusion through glass is negligibly small. In the case of metals, hydrogen has the highest diffusion rate of all the common gases; this rate increases in the order, iron, nickel, copper, platinum, silver, molybdenum. The mechanism of diffusion is different from that with glasses, the rate being more strongly dependent on the concentration gradient of the gas atoms within the metal. The approximate temperatures at which P (in the above units) has a value of 3×10^{-9} for the systems H_2/Fe , N_2/Fe , H_2/Ni and H_2/Cu are, respectively, 220, 820, 300 and 600°C . Here again, therefore, diffusion is not likely to contribute seriously to leakage problems except at high temperatures such as may be used with vacuum furnaces.

For rubber gasket materials, Dawton⁴ states that the permeability is a more serious problem with continuously pumped systems than is leakage. The data given corresponds to a permeation rate for natural rubber at room temperature of about 10^{-6} c.c./sec./ cm^2/mm . thickness/mm. Hg pressure difference, that for the synthetic neoprene being some 70% less, values which are considered too high to permit the use of these materials as bellows or diaphragms in high-vacuum systems. It was not found possible to improve the resistance to permeation by coating the rubbers with vacuum greases or paints. It has also been suggested that the silicone rubbers have a tendency to simulate diffusion through very fine capillaries.⁷

Leaks through permanent seals and joints

For present purposes, seals and joints may be classified as permanent, the object being permanent constructional connection of sections of similar or dissimilar materials, and temporary or demountable, the object being to provide for ready dismantling of sections or entry into the system. Examples of permanent ones are welded or soldered joints connecting metal parts, glass-to-metal and ceramic-to-metal seals to provide electrical lead-ins or otherwise, other dielectric-to-metal seals

and ceramic-to-glass seals to provide furnace tubes, for instance.

Primarily due to the interests of the incandescent lamp, radio-valve and x-ray tube industries, a wealth of information on glass-to-metal seals has been accumulated over the past 20 years.^{16, 18} As electrical lead-in wires, platinum or copper-clad wires are suitable with soft glasses, tungsten and molybdenum with hard glasses, and molybdenum (ribbon preferably) with silica. Large-diameter tubings can be jointed using the Housekeeper feathered seal, copper being best with soft glasses, and specially developed nickel-iron alloys such as *Fernico* or *Kovar* with hard glasses. For an efficient leak-proof and stain-free seal, the coefficients of thermal expansion of the glass and metal should be as close as possible. The metal oxide should also be capable of 'dissolving' in the glass so that a gradation from metal to thin film of metal oxide to oxide/glass composition to pure glass is obtained. Full details are available on the matching of glasses and metals and on the necessary pre-treatment of the metal surface which, if followed, will give absolutely leak-proof seals. Another technique is to fire on to the glass surface a coating of a metal 'paint' such as silver, platinum or molybdenum, then solder the metal part to this thin coating. This method is also satisfactory for sealing ceramics to metals. Glass-to-metal seals may break down at high applied voltages such as are used in nuclear physics systems, and ceramic insulators sealed to metal through rubber gaskets are considered preferable, seal diameters up to 1 ft. diam. being possible.² Mullite and zircon porcelains can be directly butt-sealed to Pyrex glass to give strong furnace tubes capable of maintaining vacua of better than 10^{-6} mm. Hg at 1,175°C., where quartz tubes become porous.¹⁷

Defective welding on vacuum vessels is a common source of leaks,^{2, 18} not only at the welds themselves, but also through cracks caused in the nearby body of the vessels. Such cracks may be detected by x-ray examination. To facilitate leak detection and repair, Bush² strongly recommends a double weld, one on the vacuum side, the other on the air side, with a series of cross dams shutting off the space between the welds into separate compartments each having a plug hole through the outer weld. The intervening space can then be evacuated in sections to test for leaks, which can be soldered when found. Bush also states that the *Fernico* and *Kovar* alloys may be rendered porous if silver solder is used on them.

Demountable joints^{2, 4, 5, 18}

For all-glass systems ground joints lubricated with grease or sealed with a sealing compound may be used, but these are limited in size. Relatively large joints are necessary for connecting up pumps, pipelines and vacuum vessels in metal

systems, or attaching gauges and providing port holes. Flanges are best for such purposes, a gasketing material being used between the two surfaces and the necessary pressure being applied by bolting the two flanges together. Dawton recommends square cross-sections for metal flanges and circular for glass ones in that the vacuum performance is then better, although the square cross-section flanges tend to split around the edges at high clamping pressures. Bush states that smooth metal surfaces allow lighter loadings to be used.

Preferably the flange should carry a double groove, which may be adapted to allow the intervening space to be evacuated to test for leaks as with welded joints. The grooves should be so arranged that there is maximum metal-to-metal contact, and minimum gasket-to-vacuum or gasket-to-air contact, thus minimising the gassing or permeability effects previously mentioned. Rectangular grooving, though more expensive, is better than circular grooving. Stops should be suitably placed to prevent excessive pressure on the gasket itself, as this may lessen its useful life. The experimental tube in Fig. 1, used to investigate the vacuum properties of gasketing materials, illustrates some types of flanges.⁵

It is generally agreed that metal gaskets give the most vacuum-tight seals: Lead, silver, copper, aluminium and tin are the most suitable metals. However, these require higher clamping pressures than do rubber, and therefore stronger and more expensive flanges, which may be unwarranted except where the flange attains temperatures above 150°C. There is some difference in opinion regarding the relative merits of rubber, neoprene, butyl rubbers, *Silastic* and *Teflon*, and there is no data truly comparative for all. All tend to flow under stress and are subject to permanent set under load, depending on the temperature. Bush states that synthetic rubbers tear more readily than natural rubber, but swell less in contact with oil. He gives as maximum safe temperatures for use, 90 to 100°C. for neoprene, 120 to 125°C. for natural and other synthetic rubbers and 200°C. for *Silastic*. He also states that neoprene was the most widely used material for all-round performance until replaced by a special gasketing material composition based on *Hycar* OR-15 and *Gastex* carbon with suitable depolymerisation inhibitors and plasticisers.

Taps, valves and movable seals^{2, 18}

Again on all-glass systems ground-glass taps suitably lubricated are limited in size. For industrial applications, valves of large bore are required—from 4 in. to upwards of 3 ft. in diam. The general principle is to have a gasket mounted on a movable shaft passing through the wall of the apparatus, and to press this gasket firmly on a valve seat within the vacuum chamber itself, as illustrated in Figs. 1 and 2. A prerequisite is a vacuum-tight seal for the shaft.

In the case of all-glass apparatus, move-

ment within the vacuum can be obtained by magnetic forces, using a movable iron part within the system and an externally applied field. Metal bellows or sylphons may also be used with glass or metal systems (Fig. 1), being joined by glass-to-metal seals or welding respectively. These also allow some lateral movement, but are difficult to replace should leaks develop in the bellows itself. The other common method is to use a Wilson-type seal consisting of a shaft passing through a doubly gasketed cell or sealing gland employing rubber gaskets suitably lubricated. The external atmospheric pressure maintains the rubber in contact with the shaft which, suitably lubricated, may then be used to impart sliding or rotary motion. Bush recommends silicone grease as lubricant, and careful control of temperature to prevent undue fluidity of the grease giving poor contact, and to lessen permanent set of the gasket rubber, for which neoprene or butyl rubber is recommended. However, Dawton considers a high-vacuum oil to be better than a grease for operation at higher temperatures. With reciprocating shafts he found a leak rate of 0.01 micron l./cm. shaft stroke for a $\frac{3}{8}$ -in. shaft, 85% of the leak occurring on the inward stroke; the rate for the stationary shaft was about one-tenth of this, and the gas appeared to be condensable vapours from the oil.

It is possible to convert standard refrigeration-type valves to vacuum valves economically, but cast-body valves should be first tested for porosity. The remarks on gasketing materials for flanges apply equally well for the gasket on the valve seat, but it must be remembered that the vacuum seal at the gasket must usually be capable of withstanding pressure differentials operating from either side. Where this is not so, a gate or disc valve may be used, relying on the atmospheric pressure on one side for effective closure; valves up to 30 in. diam. have been made using this principle, and are especially suited for shutting off diffusion pumps from the air. A number of vacuum valves of different types are now available commercially.

A number of precautionary measures of use in lessening the incidence of leaks have been mentioned, but efficient control of leaks can only come with efficient methods of leak detection. These methods are intimately associated with the performance of a vacuum gauge of some type. It is therefore proposed to treat both these aspects of vacuum technique in a later review.

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OILS and FATS

Extraction, refining, hydrogenation, fat splitting, soap making

By M. K. Schwitzer, A.M.I.Chem.E.

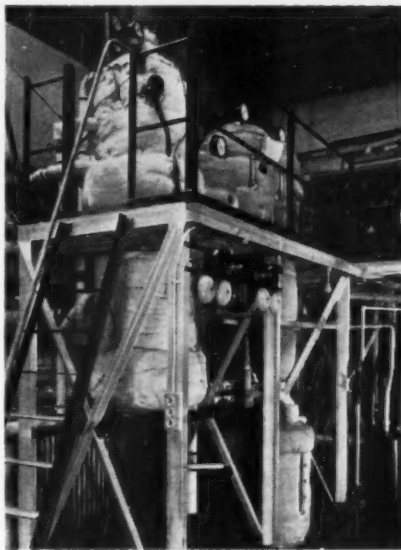
World production

RAW materials and staple foods in short supply during the war became more plentiful in recent years until present stockpiling started another period of scarcity. Oils and fats, however, are an exception; they have been scarce for the last decade. One of the main reasons for this is that the Eastern producing countries are keeping more of their supplies for their own use. Allied to this is the fact that the world population has increased by about 200,000,000 in the last ten years. Although production of vegetable oils in 1949 was more than 15,000,000 tons, compared with an average of 13,500,000 tons in 1934-38, world exports fell to 3,029,000 tons in 1949 from an average of 4,176,000 tons in 1934-38. There was only a slight improvement last year on the 1949 figures. One of the countries where vegetable oil consumption has increased is the United Kingdom. Margarine production here was 420,000 tons in 1949 against 208,000 tons in 1938, partly because of the increased use of whale oil. In the U.S.A. only 384,000 tons of margarine were produced in 1949. However, American cooking fat production in 1949 was 662,000 tons in 1949 against only 217,000 tons in the same year in the U.K.¹ But in many countries, particularly in Europe, fat consumption is well below the pre-war level.

There is little doubt that the shortage of raw materials and the necessity to reduce waste has led to new techniques in oil and fat processing. Brandt² asserts that in recent years 'technological progress has had a powerful impact upon production, processing, utilisation and consumption of fats. It leads to shifts which sometimes come slowly and sometimes abruptly.'

Oil extraction

A number of studies on oil extraction have been published. Spadaro *et al.*³ dried cottonseed flakes at temperatures up to 240°F. for 3 hr. and then extracted the oil with a solvent. Drying flakes at these temperatures had only a negligible effect on the colour of the refined and bleached oil. But properly dried flakes gave optimum conditions for solvent extraction such as percolation rate, extractability of flakes and minimum fines. The relatively high temperatures did not affect the protein solubility of the extracted meal which is desirable. Parkin⁴ gives an account of the solvent extraction of drying oils. Lower solvent losses and lower solvent ratio are claimed for batch extraction than for con-



(Photo: Bamag, Ltd.)

Continuous fat splitting plant.

tinuous counter-current methods. 'The efficiency of extraction is no doubt equal to or possibly even slightly better for the batch; however, to weigh against this a large amount of labour is required for batch extraction.' Although solvent extraction is encroaching more and more on pressing methods, Dunning⁵ has some pertinent things to say about mechanical extraction methods. Modern American expellers and screw presses give oil cakes with very low oil residues.

The continuous extraction of oil from rice bran has been reported for the first time. A plant built by Allis-Chalmers has a rated capacity of 50 tons/day and produces a crude oil containing 3 to 6% of free fatty acids.⁶ Beckel *et al.*⁷ have been granted a further patent on their alcohol extraction process; 95% ethanol is used to extract soya bean flakes containing less than 3% moisture. The miscella is cooled to separate the oil from the solvent, and the supernatant liquor recycled to the extraction column. Recycling can be repeated almost indefinitely without the addition of fresh solvent. Among several patents covering solvent extraction design, Straight's⁸ continuous counter-current extractor and Beeson's⁹ vibrating draining device for removing solvent faster from the meal may be instanced. Karnofsky¹⁰ describes the new horizontal solvent extractor made by the Blaw-Knox Co. There are now two of these extractors

installed, producing a meal containing only 0.5% of oil. The solvent ratio is said to be 0.8 lb. of hexane/1 lb. of flakes and solvent losses are 5.5 lb., against the more usual 14 lb./ton of material treated.

Solvent extraction is spreading also to the less industrialised countries. Parekh¹¹ recommends for India a combination of pre-pressing and solvent extraction when working on groundnuts. At present some 135,000 tons of groundnut cake are produced there annually, of which practically all is used as fertiliser. If properly solvent-extracted the meal could be used as cattle feed and human food.

Fish oils

Although world production of fish oils is small compared with vegetable oil production, it is nevertheless an important industry, particularly in Norway, Iceland, North Africa, the U.S.A. and Japan, and much technological progress is being reported. World production in 1949 was 292,000 tons, against an average of 460,000 tons in 1935-39. In the U.K., 1949 production of herring oil was 2,330 tons, against 1,100 tons in 1948. Apart from 150 tons, the whole of the 1949 production was used for edible purposes. The final figure for 1950 is not yet known, but is not expected to exceed the 1949 level.

At Louisburg, Canada, a new \$3,500,000 fish reduction factory is being fitted largely with Farramatic equipment shipped from the U.K. It is rated to process 66,000,000 fish annually.¹² At the other end of the Commonwealth, in New Zealand, liver oil factories were established in Auckland and Wellington. Annual production is 30,000 gal. of liver oil, of which 17,000 gal. are exported.¹³ A report from Hull states that the 750-ton *Princess Elizabeth*, a floating factory trawler, has recently sailed for her maiden voyage. She is the first vessel of its type to carry a complete factory to convert daily 10 tons of offal into 2.5 tons of meal. There is also a cod-liver-oil extraction plant on board.¹⁴ The American fish factory ship, *Oceanlife*, is equipped with a filleting machine and refrigeration unit which freezes fillets at -32.2°C.¹⁵ The plant for turning fish offal into meal and oil comprises a disintegrator, digester-cooker, press, centrifuges to separate oil from stickwater and a meal drier.

Ziembra¹⁶ gives a detailed account of the processing of stickwater as practised at the factory of the Associated Fish Products Co., Eastport, Me. Acidulated stickwater is purified, centrifuged and then concentrated to give 'condensed fish solubles.' American condensed fish solubles obtained from the stickwater from pilchard processing were investigated in England by Coles and Preston.¹⁷ This syrupy material contains 50% solids; the vitamin B₁₂ content of the solids is 1 to 4 mg./g. It is now generally held that associated with this vitamin complex is an unidentified 'animal protein factor' (A.P.F.) which is absent in vegetable proteins. When fed to chickens

this factor results in better hatchability, faster rate of growth and improved egg laying.

The following amounts (in millionths of a gramme per lb.) have been quoted for vitamin B₁₂ potency: meat meal 36, fish meal 110, crab meal 150, liver meal 235 and fish solubles 400.

A new herring oil production process has been patented by the Norwegian Notevarp.¹⁸ Fish waste is dried to 40 to 55% moisture content, then heated and pressed. A good-quality oil is obtained and losses are at a minimum. The press residue is further dried.

Fat refining

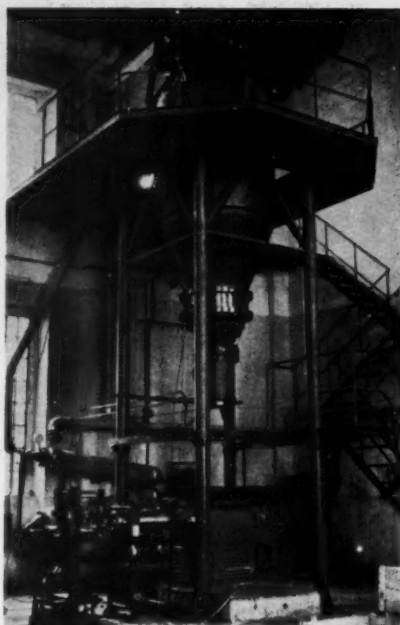
A new departure in refining fatty oils is revealed by Unschuld.¹⁹ Soya or a similar oil is passed at reduced pressure between electrodes at a voltage of 500 to 2,000 and a frequency of 500 to 4,000 for 30 to 60 sec. Fatty acids and phosphatides are thus removed, leaving a deodorised and decolorised oil. Rini²⁰ reports that the addition of an inorganic phosphate to the oil during neutralisation gives an improved product. Ramaswami²¹ shows, by means of most careful and detailed calculations, that continuous centrifugal refining plant gives refiners a considerable advantage, due to lower oil losses. A typical modern American oil-processing plant, comprising continuous refining and bleaching, batch hardening, continuous deodorising and shortening and margarine manufacture, is described by Shearon *et al.*,²² while German kettle refining and hardening operations are discussed elsewhere.²³

Hydrogenation and hydrogenolysis

Andre²⁴ traces the history of oil hydrogenation from the theoretical work of Raoult, Sabatier and Senderens. Oil hydrogenation was first patented in 1902 by Normann. Leprince and Siveke in Germany and Crosfield & Sons Ltd. in England were the first to put the new technique into practice. By 1914, there were 24 hydrogenation factories in Europe and, by 1926, 62. The U.S.A. had 15. Lorry²⁵ comes to the conclusion that hydrogenation methods employing gas circulation give best results, as the product is hardened more selectively. Halls,²⁶ in discussing synthetic waxes, gives relationships between the iodine values and the melting points of a number of fats hydrogenated to different degrees. Natta²⁷ discloses a process which permits the production of unsaturated alcohols from unsaturated acids by hydrogenation at 240 to 280°C. and 100 to 300 atm. pressure. Copper and cadmium oxide catalysts are used.

Margarine and antioxidants

A new continuous margarine plant has recently been put into operation in Drogheda, Eire, by W. & C. McDonnell Ltd., a Unilever company.²⁸ It is one of Europe's most modern margarine factories. Hardened oil is piped through glass lines



Mazzoni continuous soap making unit, showing vacuum vessel on top and plodder underneath.

(From 'Continuous Processing of Fats' by M. K. Schweitzer, A.M.I.Chem.E., recently published by Leonard Hill Ltd.)

to the continuous margarine machines, which have a rated throughput of 250 tons/week. An apparatus²⁹ for plasticising margarine continuously extrudes it through a tunnel across which is a cutter rotating about an axis at right-angles to the direction of travel of the margarine. The aromatisation of margarine or butter, without using a microbiological starter, by adding a mixture containing diacetyl, lactic acid and ethyl or a similar alcohol, is disclosed in another British patent.³⁰ In an American patent³¹ a method for colouring margarine by means of injecting pigment by a hypodermic needle into the interior of the package is described.

A summary of present trends in the application of antioxidants for edible fats is given by Sisley.³² Antioxidants may be divided into those obtained from vegetable sources, synthetic materials, synergists and lecithins. At least 100 antioxidants and stabilisers for edible oils have been described in the past year.

Fat splitting and fatty acids

A German continuous fat-splitting plant which allows a 100% split by recycling unsplit material has been described by Konstandt.³³ Glycerin concentration is 80 to 90%. Production costs are about 1.20 D.M./100 kg. of fat. Capital investment for a 1,000-ton-p.a. plant is about 75,000 D.M. The advantages of introducing fat-splitting technique into India have been studied.³⁴ The split acids have many industrial uses, and glycerin is a valuable by-product. A new American factory in which fat splitting and fatty-acid distillation is carried out in one continuous

operation was put on stream early last year.³⁴

A process for separating fatty acids has been patented in the U.S.A.³⁵ The mixture of split acids is cooled and treated with a solvent at such a temperature that the higher melting fraction is insoluble in the solvent. The undissolved fraction is then separated from the dissolved fatty acid. Separating fatty acids by solvent crystallisation has been patented also by Emery Industries Inc.³⁶ According to this method, crystals are separated from a solution of cooled, mixed, saturated and unsaturated acids in a solvent. The presence of 0.2% to 3.5% of neutral oil facilitates crystal formation. Blowing air into the stock for several hours has a similar effect. The first plant of this type in England was started up recently.³⁷

Continuous soap-making

Although there is no justification yet to declare the centuries' old soap kettle as obsolete, there is a distinct trend at present towards the continuous automatic soap-making technique. Reviewing modern continuous soap-making methods, Charlton³⁸ states that processes in which the fat and caustic lye are sprayed into a chamber are unsatisfactory. Fat splitting, followed by neutralisation of the fatty acids, omits the removal of the nigre, while the centrifugal process does not allow the 2- to 3-day period when the soap cools from 212 to 170/150°F. and changes its physical characteristics. Lachamp³⁹ describes a French method which is based on increasing the contact area between the fat and the alkaline lye, by producing an emulsion of the water-in-oil type.

A new Italian apparatus⁴⁰ for rapid mixing and producing soap in one continuous operation has been described. The plant consists essentially of a vacuum chamber from which soap powder falls into a plodder.

Another way of producing soap continuously⁴¹ is to dissolve fat in kerosene or a similar inert solvent and reacting it at 100 p.s.i. and 400°F. with a caustic lye. The reacted mixture is sprayed into vacuum. Soap powder is collected and the vapours pass into two condensers for glycerol-solvent and water respectively. The yield of free glycerol is 96.6%. Yet another continuous soap-making method is the Sharples process, using centrifugal separators.⁴² The first of these to go into operation in Europe was at a Nijmegen (Holland) factory. To treat 10 tons of fat only 1,000,000 calories are needed, compared with 13,000,000 previously. From this fat 15 tons of soap are obtained, against 12 with the old process, and 900 kg. of glycerin against 600 kg.⁴³ The adoption of the continuous soap manufacture in India and the disadvantages and merits of a number of processes is discussed by Kodavanti.⁴⁴

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Rubber to Metal Bonding

RUBBER has for a long time been extensively used in the food processing and chemical industries as, for example, in the gaskets of milk coolers and also for lining tanks and other pieces of plant. In the past it has been the practice either to spray thin coats of rubber paint on the metal or to glue sheets of rubber into position by means of adhesives, which usually consisted of a solution of rubber in suitable volatile solvents. Such adhesives do not provide a very strong bond between rubber and metal and, when tested to destruction, more often than not it is the glue rather than the rubber that fails. This weakness can be eliminated by adopting phenolic resin adhesives. It may even be that in making chemical plant there would be advantages in having available sheets of metal to which rubber had already been bonded rather than in constructing the plant first and lining it subsequently. This may be so particularly since these sheets will withstand quite drastic forming operations without damage to the bond.

As long ago as 1882, Charles Woodward took out a patent for improving the adhesion to rubber of 'glue, mucilage, paste, varnish, gums, resin, cement or other adhesive compounds or materials...' by treatment in concentrated acid, and it is interesting to know that however effective the treatment is with 'glue, mucilage, etc.', it is certainly very effective with some synthetic resin adhesives. The most suitable treatment depends to some extent on the rubber to be bonded, but for most natural rubbers, 4 min. exposure to concentrated sulphuric acid (specific gravity 1.84) is a satisfactory treatment. When bonding synthetic rubbers such as *Buna S*, *Neoprene* or butyl rubbers the time of exposure to the concentrated sulphuric acid should be doubled, or concentrated nitric acid used instead. Unfortunately, silicone rubbers cannot be bonded to metal by means of this adhesive, even after treatment in strong acid. All rubbers must, of course, be washed thoroughly in running water and then dried after treatment in the acid.

The effect of the concentrated acid is referred to by rubber technologists as 'cyclising.' The result is to make the rubber a little harder and to cover it with fine cracks, usually called 'crazing,' which are only noticeable when the rubber is distorted. Chemically, the change appears to be the conversion of the rubber to various isomers with superior adhesive properties.

If the 'cyclising' is unnecessarily prolonged, the surface hardening will be very marked and the rubber may be made unsuitable for use. Care must therefore be taken to avoid this happening and, for a similar reason, it is sometimes desirable to spread the acid by felt only on those surfaces of the rubber which are to be bonded, rather than to immerse the whole of the rubber in a bath of acid.

The treatment of rubber in concentrated acid only requires special plant if large numbers of components are to be bonded. For a wide variety of small parts, the acid may be contained in a glass vat or in one of the shallow enamelled dishes frequently used for the development of photographic prints. It is only worth installing a lead-lined bath for sulphuric acid if a very large volume of work is envisaged.

The bonding of rubber to metal, using the *Redux* process developed by Aero Research Ltd., is described by C. J. Moss in *British Plastics*, January 1951. After washing and drying the rubber, a thin coat of *Redux* liquid phenolic resin must be applied by brush. Since this resin must wet the metal surfaces, they must be thoroughly degreased and then sanded, shot-blasted or cleaned chemically, and then the liquid resin applied by brush, followed by dipping the coated surfaces in the polyvinyl formal powder. The parts are next clamped together with a pressure of about 50 lb./sq.in. and stoved at about 140 to 150°C. for 20 min., additional time being allowed, of course, for the heat to penetrate to the glue line. This temperature and pressure are sufficient to cause the powder to flow and the resin to cure.

Compared with the difficult and costly brass-plating process, the 'cyclising' of rubber in concentrated acid is a simple matter. The bonding of components, using clamps and an oven, often requires far less expensive equipment than does the vulcanising of rubber in contact with brass-plated metals. Despite this, it will be readily appreciated that the design of many components embodying rubber-to-metal bonds is such that it is more convenient to vulcanise the rubber *in situ* rather than first moulding the rubber and then bonding it afterwards.

Recent publications

Complete chemical plant. 'When Planning Complete Chemical Plant' is the title of a new folder by Bama Ltd., which summarises the complete installations supplied by that company to some 25 countries, including the U.S.A. Photographs of nitric acid plants in England and Spain, of a solvent extraction plant in South America, of a vegetable oil extraction and processing plant in Syria, a hydrogenation plant in India, an electrolyser in Eire and a continuous fat-splitting plant are among the many illustrations. The folder is in French and Spanish, in addition to English.

Oil refinery and process pumps. The many applications of pumps in oil refineries and chemical works are outlined in a well-illustrated leaflet recently published by Sigmund Pumps Ltd. Pumps are available for handling liquids at very high pressures and up to 900°F.

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Manufacture of Alginates

The existence of alginic acid in materials of plant life, particularly seaweed, has been known for some 70 years as well as simple and effective methods of extraction but, even now, it is doubtful whether 2,000 tons are being produced annually, in spite of its many proven applications and the abundant supply of raw materials. Here is a summary of a paper read by C. W. BONNIKSEN before a recent meeting of the Chemical Engineering Group of the Society of Chemical Industry. He describes the process and plant for the manufacture of alginates from seaweed as well as the inherent difficulties hampering its commercial exploitation. Some interesting applications are discussed.

CELLULOSE, pectin and alginic acid form the principal structural materials of plant life. They are polymers of glucose, galactose and mannose units respectively and so are similar in chemical constitution. All seaweeds contain alginic acid. Most species are quick-growing perennials, some are annuals, and obviously the total tonnage is to be measured in millions of tons annually. The raw material potential is so large that its availability for industry has often been taken for granted and herein lies one of the reasons for the abnormally slow growth of the alginate industry. The seaweed is rarely present in sufficient abundance in any one locality to justify the capital outlay necessary for its economic gathering and subsequent treatment, and the alternative of widespread gathering for transport to a factory breaks down on transport and labour costs alone.

So long as a factory's requirements of wet weed amount to no more than 2,000 tons p.a., its needs can be easily met. Even in these islands there are a dozen places where such quantities can be gathered cheaply and regularly. The same remarks apply to much of the European coastline, Norway and Brittany in particular. A proper appreciation of this observation, which probably applies throughout the world, is of fundamental importance for the continued growth of alginate manufacture into a significant industry.

Any estimate of world quantities must, of course, be sheer guesswork; attempts at local estimating have proved wildly inaccurate and very misleading. It is, nevertheless, clear that the total must be large and of considerable economic potential.

Harvesting seaweed

Before and during the early part of the war the practicability of the more obvious methods for gathering the laminarias was tested privately by the author. The most promising method both then and now was suggested by D. H. Cockburn and tested by J. F. Williams in 1941; it has come to be known as the 'cut and suck' method. A tube, on the suction of a large capacity centrifugal pump operating on a small vessel, is trailed among the weed, whether it be a floating or sinking species. The suction persuades the fronds of the weed to enter the tube, where they encounter a rapidly rotating cutter. The resulting weed fragments rise up the tube with the

water and are separated by wire screens or trommels placed above the vessel's hold. The subsequent handling of the weed from boat to shore is also accomplished by pumping.

However, wet seaweed is an expensive material by any method of gathering, and a would-be alginate manufacturer must pay careful attention to his factory siting and his scale of operation if he is to achieve a cheap product.

Factory siting

A chemical engineer will perceive at once that an alginate factory is ideally sited as close to a weed bed as possible, so long as the weed supply position remains in the condition described. But he will then recall that his factory cannot be very large, and most likely will be situated well away from industrial areas and their facilities and, worse still, will often be operating in remote and inhospitable parts of the world. It is very fortunate, therefore, that the actual extraction process is of no great complexity.

An algin factory must have access to an ample supply of not too hard water, zero hardness ideally—80,000 gal. are required for the manufacture of 1 ton of product. Its power requirement is 3 kVA/lb. of product, including process heating.

Process labour amounts to 150 man-hours/ton of product at a production rate of 1 ton product weekly, and is the only figure materially affected by the scale of operations.

There must, of course, be labour available for weed gathering and harvesting. The requirement varies a good deal according to the weed bed and only a rough guide can be given here. One man will be fully occupied for a month in gathering and preparing for transport to the factory enough weed to manufacture 1 ton of product.

Wet weed cannot be transported over long distances, not only because of the weight, which often exceeds 20 tons, for the production of 1 ton of product is high, but also because it deteriorates seriously and at an increasing rate a day or two after collection. The problem has been thoroughly explored, however, and wet weed has been successfully processed, though at increased cost, even after a week in a ship's hold or in a warehouse.

It cannot be stored for a long time and so the supply position, if dependent on such methods, is precarious. To over-

come this difficulty, essential in a factory which cannot be supplied from local resources, various methods have been tried for preserving the weed, among them storage in 1% formaldehyde solution, dosing the weed heavily with sulphur dioxide, either on the beaches or on arrival at the factory, and air drying. Except in favoured climates, the last method is costly, and sometimes impossible. Nevertheless, it is practised today on a considerable scale.

Extraction process

The few manufacturers of alginates are naturally reticent about the details of their process steps, but a study of the patent literature and the very few available publications show that they all follow the pattern advocated in 1883 by E. C. C. Stanford, who first identified alginic acid and extracted it from seaweed. In this the seaweed is macerated with dilute sodium carbonate and the resulting pasty mass diluted with sufficient water to make practicable the separation of the insoluble matter remaining. The resulting liquor contains most of the alginate originally present in the weed, and it can be recovered by stirring into the liquor mineral acid, or solutions of most metal salts, whereupon gelling and subsequent syneresis of the alginate occurs. The flocs of jelly are strained from the liquor, further consolidated by pressing and sometimes dried.

These very simple operations are today performed mechanically and linked together into a continuous process of very low total cost, an achievement taking the industry many years to accomplish. Further advancement of the industry no longer depends on effort directed towards processing but on the compliance by the industry with the facts governing their raw material supply. The correct choice of process steps and the plant to perform them depends on a knowledge of certain general principles about alginates and their properties, unknown and unsuspected in Stanford's day and for 50 years thereafter.

Alginic acid is a polymer of mannuronic acid with a fairly high equivalent weight, taken to be 194 by the industry as a whole for control purposes and commercial transactions. It swells, but is insoluble in water, and its titration curve was first determined by R. C. Rose working with F. G. Donnan in 1937. It has a dissociation constant of between 3 and 4. For the purposes of the chemical engineer it is a well-defined substance behaving as a moderately strong

acid, forming salts whose composition is indicated by its equivalent, those of the alkalis and ammonia being soluble in water and, with one or two exceptions, all others insoluble.

The acid and its salts are base exchange materials moving slowly into equilibrium with dilute solutions with which they may have to come into contact and, in doing so, exhibiting swelling and syneresis effects which must be allowed for in selecting plant. The manufacturer of alginates is usually handling both solutions and jellies of alginic acid at great dilution, 1/50 and 1/100 of an equivalent per litre being common. His reagents also are handled at extreme dilution, so that it is not surprising to find these jellies and solutions responding to very small changes in reagent composition or concentration, taking up some ions with great avidity, rejecting others, thus entirely altering the mechanical characteristics of the materials to be handled. They are all examples of membrane phenomena which one could expect to find with a colloidal electrolyte of this nature, and the simple Donnan theory helps the engineer and chemist considerably in groping through these many variables. In general, during processing, the alginates are impermeable to anions.

As an example of the improvement in processing brought about by a detailed study of these effects, in the author's experience there was a time when 600 sq. ft. of draining surface was necessary for the production in 24 hr. of 1 cwt. of (dry weight) product, while today draining has disappeared altogether as a process step.

The other point a chemical engineer must consider carefully arises from the polymeric nature of the material. Everything points to the fact that alginates occur in seaweeds as calcium alginate of very high molecular weight. By double decomposition this is converted to sodium alginate, and if special precautions are not taken, this too will be of very high molecular weight, giving rise to solutions of extremely high viscosity, or alternatively, and worse in its effects, of varying viscosities.

The remaining point to be borne in mind by the chemical engineer is the steady degradation of the polymer. This is brought about by enzyme action, fermentation, acid and alkaline hydrolysis and oxidising agents, and the rate of breakdown is increased by increasing temperature. Today this degradation should not take place by accident, but all these agencies are deliberately used by the manufacturer of alginates in a controlled manner to reduce at the appropriate time the molecular weight of his product and the viscosity of his solution.

The major applications of alginates require for their performance a product of low viscosity (10 to 20 cp. at 25°C.). Here and there applications are met with where higher viscosities are essential, but they are not the rule. It is essential to be clear

from the start which family of alginates are to be manufactured and not leave their emergence to chance.

The first essential step in alginate production is the reduction of the raw material to as small a particle as practicable. This is a very expensive operation and thorough tests on a production scale extending over long periods have been made of methods which would avoid this necessity, but without success.

Next, besides its calcium alginate, weeds contain a bewildering number of other substances mostly water soluble. For most of them no economic method is known for their recovery from dilute solutions, so, valuable though they may be—potash and mannitol for example—they are removed by a washing process designed primarily to avoid throughout the subsequent processing disturbances which would be caused by the presence of colloids other than algin. This washing step is carried out at elevated temperature for a definite time and at controlled pH value in the region of pH 3.

The emerging washed weed is now treated with sodium carbonate, preferably at a temperature high enough to sterilise the resulting solutions, though the double decomposition can be and has been successfully carried out in Scotland at temperatures approaching zero centigrade. During this digestion period the weed fragments swell enormously as the double decomposition proceeds, but they will not break up and disperse without powerful stirring. The product is a very viscous liquid which must be diluted with many times its own volume of soft water before it can be clarified. The separation of insoluble matter from the clear liquor is the subject of many patents, today however, super decanters or continuous settling devices suffice.

The resulting clear liquor is now poured into dilute calcium chloride or dilute sulphuric acid solution. The product of the reaction is, however, a heavily hydrated gel and the subsequent handling of this jelly is much influenced by the concentration of the clear liquor on the one hand and the concentration of calcium and hydrogen ions on the other. Unless extravagant amounts of chemicals are used the jellies require very special devices for their reduction to a form in which they can be pressed in roller presses or expeller presses. Calcium alginate can be pressed to about 50% dry matter content, alginic acid to 30 to 35%.

This is as far as processing need proceed on the site of the weed and, of course, the choice of plant depends in great measure on the extent of the seaweed bed. The beds are usually small, too small to support a local drying organisation, and the alternative of transporting the weed to a central drying organisation as a preliminary to algin processing is very expensive. The alternative, to extract calcium alginate, not alginic acid, on the site, raises the question of the minimum size of economic extraction plant.

The extraction plant

An alginate plant should be made up of a number of small units, each one complete in itself. At least two units should be operated and more if the extent of the weed supply permits. The amount of weed available for industry, would be enormously increased (tenfold in this country) if an economical plant could be devised to deal with around 300 tons p.a. of wet weed. There is in existence a machine of simple construction which will handle 2 tons of wet weed weekly, producing from it calcium alginate of high quality. The machine requires no skilled attention beyond simple maintenance and indeed can be left to itself for hours at a time.

Treatment of Ca alginate after recovery and manufacture of Na alginate

Unless the pressed calcium alginate is of high quality it has little value. It cannot be subsequently purified without repeating all the operations already described. It usually contains a small percentage of chalk, however, and a little colouring matter, both of which are readily removed, the chalk by treatment with hydrochloric acid and subsequent recalcification with lime; the colouring matter by the application of small quantities of bleaching powder followed by careful washing.

This bleaching operation requires much care, as degradation of the alginate takes place during it. To reduce the degradation to a minimum as little available chlorine should be used as possible and in extreme dilution. The action therefore takes many hours.

The colour of the recovered calcium alginate is very largely governed by the weed species and the conditions under which it is stored. The practice of air drying the weed and storing it for long periods while awaiting kiln drying is bad from this point of view. All species of weed when processed as freshly gathered weed yield a calcium alginate requiring very little chemical bleaching.

During this operation calcium alginate looks and handles like wood pulp, and the precautions to be taken are the same as with wood pulp, with the exception that the presence of calcium salts is of no disadvantage with calcium alginate.

If alginates were more plentiful and cheaper this calcium salt would satisfy the requirements of most of its applications. Today, however, the bulk of the alginates produced are marketed as the sodium salt. Almost invariably the first step is the removal of all calcium from the alginate by washing with hydrochloric acid; wet calcium alginate pulp is stirred with half normal acid for 10 min., allowed to settle and the supernatant liquor decanted, the operation being repeated many times until the liquors decanted are free from calcium. The resulting alginic acid is then washed with soft water. Counter-current methods are, of course, used in practice, requiring for complete removal

of the calcium up to 10 changes of liquor. The treatment is best done mechanically throughout, the solids moving with constant agitation against the stream of liquor. There are many standard lixiviators available and most are satisfactory, provided they do not incorporate straining devices, which would soon become clogged by swollen alginic acid. The emerging alginic acid is in the form of a highly swollen pulp which loses its water with greater difficulty than calcium alginate, although it can still be handled by press rolls.

Thereafter there are a number of methods open for carrying out the simple neutralisation of the alginic acid, with sodium carbonate, necessary to convert it to the sodium salt.

Finally, it should be mentioned that where an algin manufacturer is working for sodium alginate only he has sometimes avoided all the steps in the process for recovering the alginate as its calcium salt by adding to the clear liquors up to their own volume of ethyl alcohol, or evaporating the liquors, or again partially evaporating the liquors and then adding alcohol. Under these circumstances, sodium alginate of high quality is thrown out of solution and requires very little subsequent dressing to prepare it for the market.

Applications

In everything but supply and cost, calcium alginate can compete with starch, casein and gelatine in many of their industrial uses such as a substitute for casein and starch in paper coating, casein in water paints, starch and gelatine in textile sizing and starch in the clarification of coal-washing effluent.

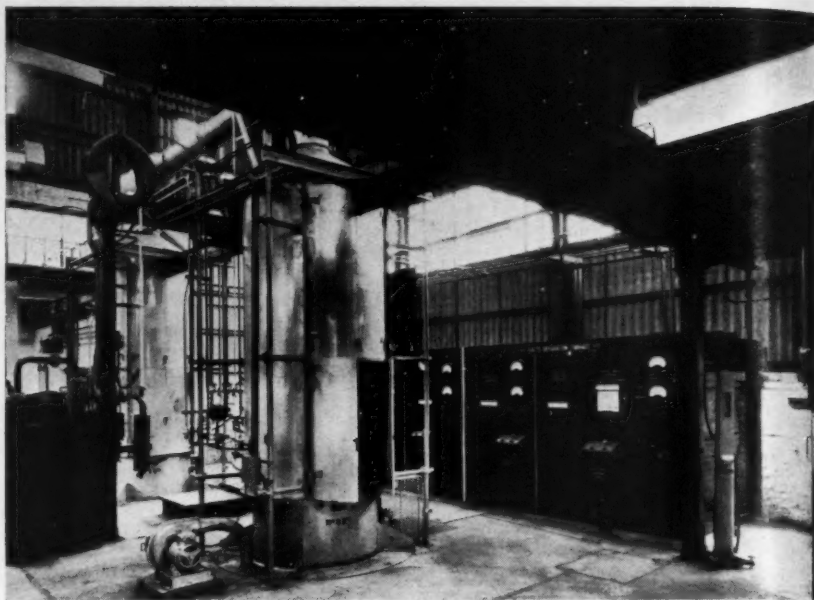
The very high viscosity of a solution of sodium alginate, especially one prepared from an alginate of high molecular weight, is utilised in the preparation of cosmetics and pharmaceuticals, including toothpaste and skin preparations. It can be used as a 'thickener' for *Teepol* solution and as a creaming agent for latex.

A high proportion of the alginates produced in the U.K. and U.S.A. are used for this purpose because sodium alginate has a remarkable potency for preventing ice crystal formation and, apparently, endows the preparation with certain desirable characteristics at the same time.

Fabrics made from calcium alginate yarn are fire-resistant and there are possibilities here, especially with the advent of the widespread use of sulphonated oil detergents, by which the fabric is not solubilised. The present fabrics deteriorate when exposed to light, however, and work will have to be done before the fabrics possess commercial value.

These are only some of the varied uses of alginates. Although this industry has not yet been fully developed and the present prices of the products do not appear competitive, their possible future value in a world short of raw materials cannot be ignored.

Zirconium Manufacture in England



Furnaces at the Murex plant used for the purification and reduction of zirconium tetrachloride. The control panel is on the right.

AS reported in our March issue, the production of zirconium has been started on a commercial scale at the Rainham, Essex, plant of Murex Ltd. Zirconium is a comparatively abundant metal; there is more of it than there is of lead, copper and zinc. However, although first isolated in 1824, it was not until 100 years later that a practical method for the production of ductile zirconium was devised.

Chemical engineering properties

The chief attraction of zirconium for the chemical engineer is its resistance to corrosion, and it is expected to replace tantalum in many applications. It may also be possible to use it with other metals to form corrosion-resistant alloys with good mechanical properties. Another interesting possibility is its use as a structural material for atomic piles.

Production process

The original method of producing zirconium compounds for the manufacture of ductile metals was discovered by van Arkel and de Boer and it is called the iodide process. It depends on the thermal decomposition of zirconium iodide at elevated temperatures. Murex use the Kroll process (see *INTERNATIONAL CHEMICAL ENGINEERING*, July 1950, pp. 315-317). It is basically different from the original iodide method and comprises: (1) Carburisation of the mineral; (2) chlorination of the carbide; (3) purification of the zirconium tetrachloride; (4) reduction of the chloride by magnesium; and (5) separation of the zirconium metal from

the magnesium chloride formed during reduction.

Zircon sand, zirconium silicate, is mixed with a form of carbon and heated in an arc furnace to form crude zirconium carbide, most of the silica being volatilised. The crude chloride is purified by resubliming in a stainless steel vessel out of contact with air and by collecting the zirconium tetrachloride vapour on a steel cooling coil at a temperature near its melting point, the chloride being obtained as a dense block adhering to the steel coil. The coil is fixed to a heated cover which floats in a lead alloy seal and, on completion of the purification, the cover with the coil and its adhering mass of chloride is lifted out and placed in the top of a similar vessel wherein a steel crucible charged with a pure magnesium ingot has already been placed. The reactor vessel is then sealed and evacuated and helium is admitted to break the vacuum. This is repeated to ensure complete vacuum. After heating, particles of zirconium are obtained in a mass of magnesium chloride. The salt and excess of magnesium are then removed by heating in a steel retort under vacuum at a high temperature. The magnesium chloride is melted and the magnesium is vaporised. The vacuum is broken gradually and the resulting zirconium sponge removed.

Although only a very slight amount of magnesium chloride is left in the zirconium it is not in a form suitable for manufacture and the sponge (small metal particles sintered together by the heat of processing) has to be melted to form a solid ingot before fabricating.

New Plant and Equipment

Materials handling equipment

A model of a materials handling unit operating in Cyprus, constructed to a scale of one-twelfth full size, as far as the stations and mechanical components are concerned, is being demonstrated by the makers, British Ropeway Engineering Co. Ltd., at the Toronto Trade Fair, May 28 to June 8. This model demonstrates the automatic handling of materials carried out without attention from operators. The material is loaded into buckets at the loading station, the buckets being fed by an automatic system of haulage on to the main track ropes of the plant and proper spacing ensured between each load. The bucket is automatically filled from a bunker by a power-operated shoot and carries its load round an angle station to the discharge terminal at the unloading end. In the actual installation it would deposit its load into the hold of a vessel at sea, but in the model the bucket discharges into a hopper, from whence by a system of conveyors the load is returned to the loading station and the whole cycle recommences.

Sleeve-packed cocks

A patent sleeve-packed cock is being marketed by Richard Klinger Ltd. which is claimed to replace small taper plug cocks for pressure gauges, air reliefs, drains, instrument connections, compressed air, gas or hydraulic controls and similar equipment.

Machined from a hot brass stamping and polished all over, this is a simplified design with a minimum of components, based on the Klinger 'sleeve-packed' patent.

The principle of the cock is that the packing consists of a renewable sleeve of compressed asbestos which is inserted in the body as a single unit. The plug is perfectly parallel and is ground to size, so that there is no risk of the cock jamming when it is tightened up. The renewable packing sleeve is made from resilient Klingerit compressed asbestos, and is built up in layers. A ridge on one side of the sleeve fits into a corresponding groove in the body and ensures that the ports are always in line.

Steel eyelets recessed in the sleeve pre-

vent the asbestos from squeezing into the ports when tightening up and at the same time prevent the passing fluid from cutting the asbestos packing. These eyelets are conical in section, so that when the bottom or top tightening nut is followed up the tendency is to squeeze the asbestos more tightly around the plug, and at the same time the eyelet is pushed towards the cock body, so that there is no risk of it scoring the plug.

Tested hydraulically to 1,000 lb./sq.in. (70.3 kg./cm.²), it is claimed to give reliable service and easy maintenance with long life under the most exacting conditions.

Layer thickness meter

A pocket-sized instrument for the measurement of non-ferrous layers on ferrous bases is now being marketed by the General Electric Co. Ltd. The instrument, weighing only 6 oz., has many industrial uses in engineering, metal finishing, etc., because it enables the thickness of paint-sprayed metal or other protective layer to be determined within a matter of seconds.

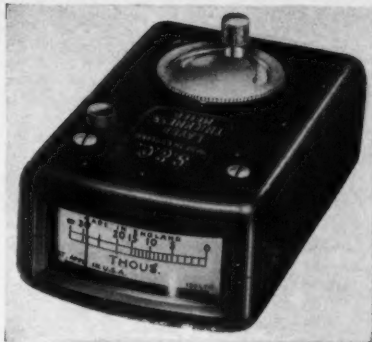
In determining the thickness of layers which are bonded to the base material the instrument is used direct, but for such substances as paper, leather-cloth, foils, electrical insulating cloths, etc., the material is tested in contact with any convenient iron or steel surface.

The instrument does not require an electrical supply and it is, in fact, a magnetic reductometer which operates on the moving-iron principle.

A shaped, moving iron is mounted at the neutral axis of a powerful permanent magnet. One end of the magnet is fitted with a spherical polepiece of hardened steel which projects through the bakelite case of the instrument.

The moving iron, which is spring controlled, has a partially suppressed zero position and takes up a position of equilibrium when the magnet and spring torques are equal and opposite.

If, then, the spherical steel tip is placed on a ferrous plate, the magnetic flux distribution will be disturbed and the moving iron will take up a new position of equilibrium. Because of the suppression of the spring, i.e. it has a small torque when the



Meter for measuring non-ferrous layers on ferrous bases.

instrument is at rest, small non-magnetic layers placed between the steel tip and the base will cause relatively large movements of the moving iron and the scale can be calibrated in thickness of the non-magnetic layer.

Besides being used for absolute measurements, the meter can also be used as a comparator or limit gauge, by setting the pointer at the desired scale point when the instrument is placed on a known thickness.

A feature of the instrument is that the pointer locks automatically, except when a small push button on the case is pressed. This enables measurements to be made in out-of-the-way places such as in the corners of tanks or the undersides of steel plates.

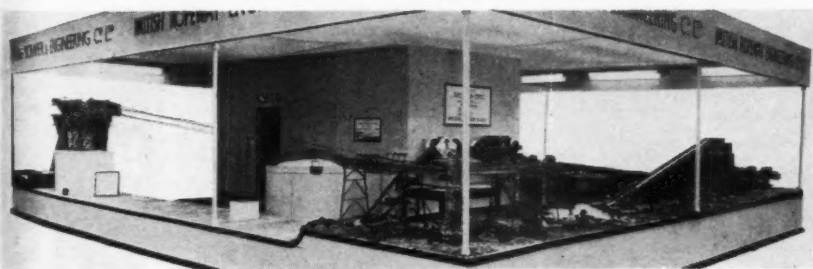
The meter is available in three single-range forms of 0 to 5, 0 to 10 and 0 to 30 thousandths of an inch.

Glass bubble-cap column

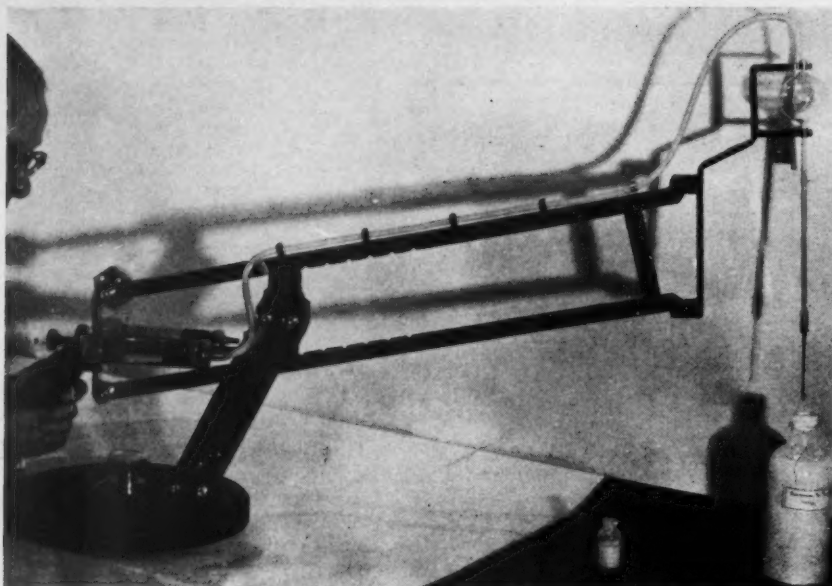
Although glass is the ideal construction material for many operations involving corrosive chemicals, until recently difficulties and cost of manufacture have discouraged the use of glass bubble-cap columns for commercial fractional distillations. The development of new fabricating techniques, however, has made the manufacture of suitable equipment economically feasible. All-glass bubble-cap columns, 4 to 24 in. in diameter and with integral bubble caps of conventional design, are now available.

J. C. Smith and E. F. Kelm describe how Cornell University studied the performance of a 6-in. eight-plate column, carrying one cap per plate, to provide basic data on such columns, in *Chemical Engineering*, 1951, 58, p. 155.

Slots in the caps were equally spaced, except that the two in direct line with the downcomers were omitted. There was no skirt clearance, each cap being integral with its plate. The downcomers were made of borosilicate glass tubing (Pyrex brand No. 7740); plates were high-silica glass (Vycor brand No. 7900). To allow for differential expansion between the plates and the downcomers, Teflon sleeves were used to hold the downcomers in place. Spool pieces were 8-in. lengths of flanged No. 7740 glass pipe, separated from the plates by



Model of materials handling unit, 1/12th full size.



Telepipette for remote handling of radioactive solutions.

asbestos gaskets. The column was assembled by use of external flanges and tie rods.

Tests were made with each of two binary systems: carbon tetrachloride (b.p., 76.8°C.) and benzene (b.p., 80.1°C.); and methyl cyclohexane (b.p., 101°C.) and toluene (b.p., 110.8°C.).

The boiler was normally charged with about 7 gal. of mixture, and the reflux surge tank was filled with material of the estimated composition of the reflux. After a heat-up period of 30 to 45 min., another hour was required before steady-state conditions were reached, as indicated by constant density of the reflux. Reflux was returned to the column about 5°C. below its boiling point. Samples were taken of the reflux, the vapour entering the column, and the liquid returning to the boiler. In some runs samples of liquid and vapour were taken from individual plates. All samples were slowly withdrawn through ice-cooled copper tubes.

Overall efficiencies were taken simply as the number of theoretical plates divided by actual plates $\times 100$.

With the system carbon tetrachloride-benzene, the overall plate efficiencies rose to a maximum of 65 to 70% at a vapour velocity of 0.5 ft./sec., and then fell. At very low velocities the contact between vapour and liquid was not very good, but there was no liquid entrainment.

With methyl cyclohexane-toluene the over-all plate efficiencies were in the 80 to 90% range, but they fell with rising vapour velocity over the whole range of boil-up rates used. Even at low vapour velocities there was considerable entrainment.

For commercial use a glass column would normally be insulated. For purposes of demonstration and instruction, however, the test column was left bare. Heat losses from the column were appreciable, result-

ing in premature condensation of vapour within the column and affecting the apparent velocity.

The all-glass column behaved very much as expected, with somewhat higher-than-usual plate efficiencies. In general, its operation was very smooth; in the few cases when it did not work well the source of trouble could immediately be seen.



Acoustic device for controlling the feed of raw coal into a tube mill. The device was fully described in our April issue, p. 158, and as pointed out, it might be adapted for controlling the milling of chemicals and minerals provided the process is continuous.

New Plant and Equipment
Fill in the Enquiry Coupon on page 240
for further details of the equipment
and plant described in
INTERNATIONAL CHEMICAL ENGINEERING.

Telepipette for radioactive solutions

A telepipette for the remote handling of radioactive solutions has been designed by Apis Engineering & Research Ltd. It has been designed so that active solutions may be dispensed from a container without exposing the operator to excessive radiation.

The pipette is more than 3 ft. from the operator, while movement of 8 in. in the vertical and horizontal planes is possible. Any standard Elliott pipette may be fitted, to a normal maximum of 50 c.c., while the capacity of the pump may be varied to individual requirements. An automatic screw adjustment is incorporated so that the pump may not, for reasons of safety, withdraw more than the capacity of the pipette; predetermined doses may also be measured by this means. Accidental discharge of the pipette during movement is said to be prevented, while the pipette doses, which are claimed to be visible from a distance, are variable according to requirements. Removable Perspex shields may be fitted to give additional radiation protection to the operator.

World News

GREAT BRITAIN

New sulphur rationing scheme

Plans to introduce a detailed system of allocation, industry by industry, for sulphur and sulphuric acid, to come into operation this month, were announced in Parliament on April 16 by the President of the Board of Trade. This will replace the rough-and-ready scheme of licensing which began on January 8.

Speaking of the grave effects upon industry of the sulphur shortage, Mr. Wilson said that the allocation of 81,000 tons of brimstone which the U.S. had made for the first quarter of this year was totally inadequate. Even with 100,000 tons the position would still be serious, forcing a cut of 20% in acid supplies to the rayon industry and similar cuts in agricultural requirements and in supplies for paint, detergents, transparent paper, etc. The superphosphates industry would remain at its present level of less than two-thirds capacity. An allocation of 110,000 would permit a reasonable level of production.

Second quarter sulphur allocation

According to a report from Washington dated April 25, Britain has been allocated 95,000 long tons of sulphur for the second quarter of this year, from a total export allocation of 250,000 tons. This compares with the 100,000 tons received in the first quarter.

Opportunities for chemical engineers

The Institution of Chemical Engineers has more than 1,000 graduate and student members, about 45% of the total membership. Last month 85 graduates and students met at a four-day conference in London to discuss chemical engineering opportunities in specific industries. Of the 85 young chemical engineers, 44 came from industry and 41 from colleges, Battersea Polytechnic having the best representation.

Informal lectures were given by leading chemical engineers on the specific opportunities of employment in the gas, metallurgical, oils and fats, plastics, low-temperature and atomic energy industries. Several of the speakers emphasised that the best opportunities for the chemical engineer were probably to be found in the marginal industries where the current shortage of raw materials means that low-grade sources have to be utilised and economic methods for processing such starting materials have to be devised.

Visits to a number of works were made during two afternoons and the students and graduates saw a variety of processes employing chemical engineering techniques on an industrial scale.

The annual general meeting was held during the convention and Mr. F. Ross, British Electricity Authority, was elected chairman of the Graduates' and Students'

Section for 1951-52, and Mr. A. H. Isaac, Foxboro-Yoxall Ltd., the Section secretary.

To complete the proceedings, a dinner and dance was held at Williamson's Tavern in the City. Dr. E. T. H. Hoblyn, Director of the British Chemical Plant Manufacturers' Association, and Mr. J. Davidson-Pratt, Director of the Association of British Chemical Manufacturers, were the principal guests. The retiring Section chairman, Mr. L. L. Gush of the H. W. Cremer organisation, presided.

A fuller account of the convention will appear in our next issue.

Current tasks of chemical plant manufacturers

The chemical plant industry is at present heavily burdened with work and to fulfil its tasks it must have the co-operation of the chemical industries and the government. This was stated by Major V. F. Gloag, Chairman of the British Chemical Plant Manufacturers' Association, at the Association's annual luncheon in London on March 29. Continuing, Major Gloag said that the chemical industries must take the plant manufacturers into their confidence at an early stage in the planning of new projects.

Major Gloag was proposing a toast to the guests among whom were Dr. F. J. Curtis and Mr. E. B. Lopker, the two Americans who, as consultants to the European Co-operation Administration, have been discussing the sulphur situation with Board of Trade officials. Among other guests were Prof. D. M. Newitt, President of the Institution of Chemical Engineers, Mr. C. G. Hayman, Chairman of the Association of British Chemical Manufacturers, Mr. S. Robson, President of the Society of Chemical Industry, and Mr. H. W. Cremer, Chairman of the D.S.I.R. Chemical Engineering Research Committee.

The question of co-operation was discussed by Sir Ewart Smith, Technical Director of I.C.I., in his reply to Major Gloag. He said that many of the chemical industry's problems were so complex that they could not be solved without complete integration at all stages. Thus, in some cases, the industry had to make its own plant. However, it did not want to do for itself what it could obtain sufficiently and at a reasonable price from the chemical plant manufacturers. Referring to research, he said that it was wrong for a firm to suppose that because it supported a research association there was no need for it to do research. It was impossible to apply work of others unless one knew something about it. Firms should let others know the results of their research and should emulate American industry where there was a great open-mindedness and an eager exchange of knowledge.

Sir Ewart said that there was illimitable scope for the chemical plant industries, but he thought the future lay with those who specialised. Here again this was something in which the Americans excelled. There was, of course, a great need for chemical engineers and it was noteworthy that in America, where for many years they have been producing 15 times more chemical engineers than in Britain, they were getting worried about the supply of engineers.

Annual Report

Membership. The report for 1950 of the B.C.P.M.A. states that membership has risen from 141 to 166 concerns. The Association aims to enrol as members the whole of the true chemical plant industry and it is now giving special attention to this project.

Research. Dr. E. H. T. Hoblyn, Director of the Association, took his predecessor's place on the D.S.I.R. Chemical Engineering Research Committee which submitted its report in October 1950. This is the first comprehensive survey of its kind.

Raw materials. There are particularly severe shortages of stainless steel, aluminium, copper and brass. The government is being urged to consult continuously with the industry in connection with raw material supplies and the method of application of such controls as may prove necessary.

Exports. From such statistics as are available, it appears that the chemical plant industry's exports in 1950 were worth £3,140,000 compared with £3,710,000 in the previous year. Members met fierce competition from American firms in overseas markets in spite of devaluation of sterling.

Officers for 1951. Chairman: Major V. F. Gloag, M.C. (Simon-Carves, Ltd.). Vice-Chairmen: Mr. E. S. Franklin (Torrance & Sons, Ltd.), Mr. G. N. Hodson (Hathernware, Ltd.) and Mr. W. J. Hooton (S. H. Johnson & Co., Ltd.). Hon. Treasurer: Mr. P. W. Seligman (The A.P.V. Co., Ltd.).

Nuclear power research

The Reactor Physics Division, which has recently been formed at Harwell Atomic Research Establishment, will act as a focus for work on the nuclear power programme covering power applications in general. Dr. J. V. Dunworth, senior principal scientific officer, Ministry of Supply, has been appointed head of the Division.

Supply Ministry controls magnesium

The Minister of Supply assumed responsibility for the supply and distribution of all virgin magnesium in the U.K. on May 1.

Deputy becomes C.R.L. director

Dr. D. D. Pratt has been appointed Director of the Chemical Research Laboratory, Teddington, after being Acting Director for some time.

Sir Robert Robinson, F.R.S., is to act

as an honorary consultant to the Director on research in pure organic chemistry.

Chemical price increases

The following increases in the prices of chemicals were announced recently:

Sulphuric acid. The Board of Trade have made an order permitting, from April 9, an increase in the maximum prices amounting to 27/6d. per ton on weak acid (77% H_2SO_4) and 33/9d. on strong acid (more than 84.02% H_2SO_4).

Glycerin. New prices were announced on April 1 by Glycerin Ltd. B.P. grade glycerin now costs from 27/5s. to 31/2s./cwt. depending on quantities. Refined pale straw industrial glycerin costs 5s./cwt. less.

Industrial alcohol. The Distillers Co. Ltd., announced new prices on April 1. Plain British Spirit (ethyl alcohol 95% Gay Lussac 66.0 o.p.) now costs from 4/5½d.—4/8½d./proof gal.

The Methylating Co. Ltd., announce that the prices of methylated and other denatured spirits have been adjusted accordingly.

Acetic acid, acetates, etc. Because of the increases in the prices of ethyl alcohol, British Industrial Solvents Ltd. have had to increase the prices of acetic acid to £109—£129/ton, acetic anhydride to £165/ton, butyl alcohol to £250/ton, acetates to £148—£310/ton, and phthalates to 2/2½d.—2/7½d./lb.

Solvents. Shell Chemicals Ltd. announced new prices effective from March 27. Methyl ethyl ketone now costs £157/ton at the 10-ton rate, delivered in the U.K. in 40/45 gal. drums, methyl isobutyl ketone £197/ton, and secondary butyl alcohol £167/ton. The price scale of each product, quantity rebates, bulk delivery allowances, and terms remain unaltered.

Calcium carbide. From April 2 the price of calcium carbide sold through the Carbide Distributing Agency Ltd., has been increased owing to the increased cost of production. Sales delivered in one consignment from factory direct to user now cost between £27 17s. 9d. and £29 17s. 9d./ton according to the grade for lots of 4 tons and over. The prices are higher for smaller consignments and there is an additional charge for delivery from warehouses.

New plant for powdered metals

A new plant for the quantity production of a high quality iron powder suitable for the economical manufacture of general engineering components by powder metallurgy, has been erected by George Cohen Sons & Co. Ltd. The range of powders includes 'Sintrex' metallurgical iron powder, 'Sintrex' electrolytic iron powder, which is widely used in electromagnetic applications such as the manufacture of transformer cores, sintered permanent magnets, etc., and 'Sintrex' grey iron powder, of which the finer mesh gradings are used as a reducing agent in the reduction of organic chemical compounds,



Mr. M. B. DONALD, M.Sc., M.I.Chem.E., F.R.I.C. who has been appointed to the Ramsay Memorial Chair of Chemical Engineering at University College, London, will take up his new post on October 1. Mr. Donald has been at University College since 1931. He was honorary secretary of the Institution of Chemical Engineers 1937-45, chairman of the Chemical Engineering Group, S.C.I., 1945-47, and he is now vice-president of the Institution of Chemical Engineers. He is also a member of the Chemical Council and a Director of the Bureau of Abstracts.

and the coarser gradings for concrete floor hardening.

Synthetic fibre development by I.C.I.

A Council has been formed to assume responsibility to the Board of I.C.I. Ltd., for the Terylene project (see INTERNATIONAL CHEMICAL ENGINEERING, December 1950, p. 558), and to co-ordinate research and development work in I.C.I. on all synthetic fibres, excluding nylon and Ardil.

Dr. A. Caress has been appointed chairman of the Terylene council, of which the other full-time members are Mr. G. F. Whitby, Dr. R. Beeching, Mr. Rowland Hill (Dyestuffs Division), Dr. E. D. Kamm (Plastics Division) and Mr. W. F. Osborne (Plastics Division). Dr. J. C. Swallow succeeds Dr. Caress as a joint managing director of the Plastics Division. Mr. E. G. Williams has been appointed to the Plastics Division Board and succeeds Dr. Swallow as Research Director.

Chemical Society officers

The Chemical Society officers for 1951-52 are: **President:** Prof. E. K. Rideal. **Vice-Presidents:** Dr. F. G. Donnan, Sir Ian Heilbron, Sir Cyril Hinshelwood, Dr. W. H. Mills, Sir Robert Robinson, Dr. N. V. Sidgwick, Prof. G. R. Clemo, Dr. J. W. Cook, Prof. D. H. Hey, Dr. R. P. Linstead, Prof. M. Stacey, Dr. W. Wardlaw. **Treasurer:** Sir Wallace Akers. **Secretaries:** Dr. H. Burton, Prof. E. D. Hughes, Dr. L. E. Sutton.

Mining engineers to meet

The 1951 Convention of the Association of Mining Electrical and Mechanical Engineers is being held at Malvern from June 25-29. Enquiries should be addressed to Mr. J. G. Leitch, Meco Works, Worcester.

New plant for oil refineries

Exceptional movements of heavy engineering equipment will be carried out during the coming months in connection with the Anglo-Iranian Oil Company's programme of expanding its refineries in Great Britain. The largest of these is the transport of a steel column 80 ft. in length and 16 ft. in diameter, and weighing 120 tons, from the Broomside Boiler Works of Marshall & Anderson Ltd., at Motherwell, to the refinery at Grangemouth of Scottish Oils Ltd. (see INTERNATIONAL CHEMICAL ENGINEERING, April 1951, p. 165). This will involve carriage over a route of 30 miles, and is believed to be the heaviest load ever moved by road. Special bogies are being designed for it, and very careful planning of the route, with due regard to such factors as corners and bridge loads, is necessary. Experience of the movement of such large loads has already been gained, since last year a column measuring 96 ft. by 13 ft. and weighing 67 tons was successfully moved from Motherwell to Grangemouth. The new column will form part of the catalytic cracking plant being installed at Grangemouth for the production of high-grade motor spirit.

The same manufacturers are also supplying a column measuring 76 ft. by 16 ft. to Anglo-Iranian's refinery at Llandarcy, South Wales. It will be moved by road to Glasgow, and then towed down to Swansea by water via the Clyde and west coast.

G. A. Harvey & Co. (London) Ltd., are also making a number of very large columns and towers at their Woolwich works. Two of these, each 106 ft. long, 12 ft. in diameter and weighing 65 tons, will be delivered by road, one to Llandarcy and one to Anglo-Iranian's new Kent refinery which is being built on the Isle of Grain at the junction of the Thames and the Medway. The Kent refinery will also be receiving by road, from the same makers, a tower weighing 120 tons early in 1952.

British Standards exhibition

As part of their Golden Jubilee celebrations, the British Standards Institution are arranging an exhibition in collaboration with industry. To be opened by the President of the Board of Trade, it will be held at the Science Museum, South Kensington, London, S.W.7, from June 18-28, 10 a.m. to 7 p.m. daily except Sundays, admission free.

The benefits derived from standards, standardisation, and simplification will be graphically presented, and each industry will show how standards have simplified production, reduced costs and maintained quality, and how in turn they have benefited the users of that industry's products.

How research at one end of the production chain and quality control at the other are linked with and helped by standardisation will also be demonstrated. Other special features will include apparatus used in testing for compliance with British Standards.

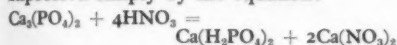
New nitrophosphate fertiliser

Many companies have been attracted by the possibilities of using nitric acid instead of sulphuric acid for the extraction of phosphate rock. Operation of the ordinary superphosphate process, replacing sulphuric with nitric acid, is unsatisfactory, however, because of the hygroscopic compounds, such as calcium nitrate, produced in the reaction. Work on nitric acid processes has been carried out by Imperial Chemical Industries at Billingham intermittently since about 1929 and during the last few years it has been possible to obtain a granulated product of satisfactory physical properties.

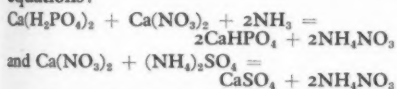
The analysis of the product is as follows:

	%
Total P_2O_5 content	15.4
Citric acid soluble P_2O_5 content ..	12.7
Nitrogen content	12.7
Water content	2.0

Imperial Chemical Industries' nitrophosphate is made by the extraction of phosphate rock with nitric acid. Ground Morocco phosphate rock and a solution of 50% nitric acid, in somewhat less than theoretical proportions, are mixed together in a corrosion-resistant steel reaction vessel in order to convert the P_2O_5 content of the phosphate rock into monocalcium phosphate. The reaction to this stage may be expressed simply by the equation:



The presence of calcium nitrate in a fertiliser is undesirable, because of the avidity with which it absorbs moisture from the surrounding atmosphere, a solid fertiliser soon changing into a fluid mass. In order to remove calcium nitrate from the nitrophosphate the reaction product is treated with ammonia and ammonium sulphate according to the following equations:



The resulting material is dried and the granules formed are screened and bagged.

The product consists of hard, stable granules with good storage properties.

Extensive field trials are in progress this season and a pilot plant is being built at Billingham.

Big coal drier for power station

A £100,000 coal-drying plant has been ordered by the British Electricity Authority from the British Rema Manufacturing Co. Ltd., subsidiary of Edgar Allen & Co. Ltd. The contract covers the complete supply of the plant with buildings and auxiliaries, at Tir John Power Station, Swansea. Anthracite duff and steam duff will be dealt with at the rate of 100 tons hr. in the 4 rapid pneumatic driers.

Acid scheme launched

A company named the United Sulphuric Acid Corporation Ltd. has been registered as a private company for the purpose of erecting and operating in this country a

plant for the production of sulphuric acid from anhydrite, deposits of which are to be found in several parts of the United Kingdom.

The project involves British Enka Ltd., the Clayton Aniline Co. Ltd., Courtaulds Ltd., Fisons Ltd., Imperial Chemical Industries Ltd., and others who, it is proposed, will subscribe for shares in the new company in the same ratio as they will respectively purchase sulphuric acid from the company. The directorate and management of the new company will shortly be organised on permanent lines.

The plant will be designed to produce some 150,000 tons of sulphuric acid p.a. and discussions are proceeding in regard to a site for the plant and other matters. The estimated cost of the capital equipment and working capital is a minimum of £3,500,000. A substantial proportion of this amount will, it is proposed, be raised in the form of loan capital.

In due course application will be made to the Capital Issues Committee for permission to raise the necessary share and loan capital.

The Board of Trade has indicated that the manufacture of sulphuric acid from materials other than sulphur is a matter of national importance, and is in accordance with current Government policy.

Cut in stainless steel supplies

On April 20, the Minister of Supply stated in the House of Commons that there was a grave shortage of nickel, tungsten and molybdenum, and consequently of alloy steels. There was an immediate need to make savings in the civil use of these metals to provide for the very heavy requirements of the rearmament programme. As an emergency measure, as from May 1, the amount of nickel supplied for stainless steel production would be cut to 70% of the 1950 level, and supplies of nickel anodes for plating would be cut to 50% of the 1950 level. These cuts would be followed by the prohibition of less essential uses of nickel. The U.S. Government had already enacted prohibitions in this field.

Instruction in welding

The British Welding Research Association is holding a summer school on "Welding Design and Engineering," at Ashorne Hill, near Leamington Spa, Warwickshire, from May 25 to June 2.

Full particulars can be obtained from the Secretary, British Welding Research Association, 29 Park Crescent, London, W.1.

Plant engineers' conference

One of the high-lights of the fourth annual conference of the Incorporated Plant Engineers will be an exchange of views on plant engineering with Mr. Richard H. Morris, an American member of the Institution. Mr. Oliver Lyle, director of Tate & Lyle Ltd., will also speak. The conference is being held at the Palace Hotel, Buxton, from May 22-24.

At the annual general meeting to be held during the conference, Mr. E. G. Phillips, will be handing over the presidency to Mr. D. Lacy-Hulbert, Chesterfield Tube Co. Ltd., after which Mr. Jack Duckham will present the Alexander Duckham Memorial Awards for 1951.

Continuous pneumatic gauging

A new application of continuous pneumatic gauging to materials in motion during production has been developed by the metrology section of the National Physical Laboratory. It has been successfully tested in a tyre factory.

Automatic gauging of wire, yarns and textile materials has been achieved by passing the material through a measuring head to which compressed air is supplied. The material restricts the outlet of the air and the varying degrees of restriction as the material moves are reflected in variations of pressure in the pneumatic system. These variations can be amplified when necessary and used to operate a recorder, so providing a permanent record of the fluctuations in size of the material being measured.

In the manufacture of tyres rubberised cord fabric is used. This material comes off the calendaring rollers at about 180 ft./min. It is 5 ft. wide and impregnated with rubber in a tacky condition and therefore difficult to measure. Variations in the thickness of the fabric can give rise to serious difficulties when the tyre is built up later, and so it is important to produce it as uniformly as possible. No satisfactory method of measuring continuously the thickness of the tacky fabric had been discovered until this form of pneumatic gauging was tried.

AUSTRIA

Liquid ammonia fertiliser

The Austrian Nitrogen Works at Linz are planning to produce liquid fertiliser from liquid ammonia. Large-scale experiments are being conducted at present in cooperation with the Vienna Institute for Agriculture. These will be continued through 1951 and should they prove satisfactory, large-scale investments for commercial production of the fertiliser will be made in 1952. The necessary machinery would have to be purchased abroad. As the works, established in 1940/44 and one of the largest and most modern of its kind in Europe, are one of Austria's most important sources of foreign exchange, it is expected that the necessary funds will be made available.

GERMANY

New crude oil refinery

A new crude oil refinery with a yearly capacity of 36,000 tons has been opened at Essen by Kleinholz and Co., in collaboration with Kraftstoff Handelsgesellschaft in Berlin. The capacity is to be raised to 60,000 tons next year. A thermal

cracking plant and a large oil depot are to be set up this year, according to the German Petroleum Information Service.

Shortage of phosphorus

A shortage of phosphorus exists in West Germany and the chemical industry is pressing for Allied permission to establish phosphorus production facilities. West Germany's requirements of thermic phosphoric acid and phosphorus are estimated at 20,000 tons annually. Due to the post-war division of the German Reich, the West German Republic has no production capacity for elementary phosphorus and chemically pure phosphoric acid.

Efforts to obtain Allied permission to establish new facilities are said to have been unsuccessful hitherto, while it has been found impossible to purchase adequate quantities abroad. The shortage of phosphoric acid is stated to weigh heavily on the match and detergent industries where 15,000 tons of phosphoric acid are annually required for the production of washing preparations alone. The lack of elementary phosphorus is also hampering the dyestuffs industries.

NORWAY

Uranium reactor ready soon

Norway's uranium reactor at Kjeller near Oslo is expected to be completed shortly, at a cost of £750,000. Only five other countries (U.S.A., Canada, Britain, Russia and France) have so far built uranium reactors. A hundred workers have been engaged in its construction and, when in operation, it will employ about 50 workers.

The reactor will enable Norway to produce radioactive isotopes which have hitherto had to be imported, and will be of great value for scientific research in the spheres of medicine, biology, chemistry, agriculture and nuclear physics.

The reactor will contain 6 tons of heavy water, costing almost £500,000, 3 tons of uranium and 45 tons of graphite. The heavy water is produced by the Norsk Hydro chemical concern at Rjukan and need never be replaced. The uranium is also produced in Norway at Evje. To protect against radioactive rays, the reactor is capped by a layer of concrete 7 ft. thick and weighing 1,000 tons.

Dutch scientists will participate in research work. According to the agreement signed by representatives of the two countries, Holland will also supply some uranium for the reactor. Norwegian research scientists express satisfaction that co-operation in the field of atomic research will be inaugurated with Holland. Odd Dahl, the engineer who has directed the construction of the uranium reactor, states: 'Co-operation with Holland will enable a far better exploitation of the reactor to mutual advantage. It is significant that two small countries are now to enter into open scientific co-operation in this important sphere in which countries have been so secretive since the war.' Mr. Dahl

added that the production of radioactive isotopes at Kjeller will probably be so large that it will no longer be necessary to import them from Great Britain or the U.S. and that there may be a surplus for export.

New plastic floor covering

A method of making plastic floor covering, incorporating rubber waste and a special black clay, has been developed by Hans Anfinset, an Oslo technician. All the materials are available in Norway. A factory at Drammen will start production this summer at the rate of 300,000 cu.m. p.a.; 2,000,000 cu.m. of linoleum are now sold in Norway annually, and it has nearly all to be imported. The new plastic covering is reported to be three times as strong as linoleum and only half the price. Manufacturers in the U.S. are reported to be very interested in making the covering there under licence. A Swedish firm has placed an order worth £50,000.

New method of utilising low-grade iron ore

Supplies of low-grade iron ore from the U.S., France and Luxemburg have been sent to Norway for trying out a new processing method developed by the Titan Co., Fredrikstad. If the tests are successful, it may mean that deposits of low-grade ore in the U.S. and Western Europe can be used for iron and steel making and thus augment the seriously diminishing reserves of high-grade ore. Tests already undertaken with low-grade Norwegian ore have proved very satisfactory, and the Titan method, described as a completely new process, has been patented in the U.S. and many other countries. The method, which has aroused widespread interest in industrial circles, involves enriching the ore to make it suitable for use in ordinary smelting furnaces.

ITALY

Montecatini's 1950 production

Production in the various branches of industry in which the Montecatini group is engaged was 22% higher in 1950 than during the previous year, while sales of the Montecatini Co. alone in 1950 amounted to approximately 76,000 million lire, according to the Chairman's annual report.

Production of pyrites in 1950 amounted to almost 900,000 tons, of which the Montecatini mines contributed approximately 750,000, slightly more than in 1949. Sulphur production was still far below the pre-war level at 213,000 tons of raw sulphur, 80,000 of which came from the Montecatini mines, where production is progressively declining. The company is, however, intensifying and extending its search for new deposits in the Romagna and Marches regions, and also in Sicily. Production of sulphuric acid last year rose further to 839,000 tons, in terms of monohydrated acid, from 808,000 tons in 1949. This total is expected to be improved again in 1951 following modifications and improvements in plant, the Chairman said.

The company is also undertaking gravimetric and geological explorations to discover new sources of hydrocarbons, both along the Adriatic coastline and in the province of Crotona, while geological studies have been carried out in the provinces of Siena and Pisa.

Production of phosphate fertilisers, and particularly of superphosphates, rose sharply in 1950, as also did consumption. Sales totalled 1,310,000 tons in the 1949-50 season, compared with 1,194,000 in 1948-49. Consumption of superphosphates reached a level equivalent to 80% of the total employed in the last year before the war—1938-39, which was itself a particularly good year for superphosphate sales. Production of primary synthetic nitrates by Montecatini reached 93,000 tons in 1950, against 76,000 in 1949.

Production of calcium cyanamide rose to 13,000 tons, from 4,200.

Italian consumption of domestically-produced copper sulphate has become stabilised at around 70,000 tons a year, requiring between 15,000 and 18,000 tons of copper. In spite of difficulties in obtaining the latter, Montecatini's production had not been affected to any appreciable extent.

Better supplies of electric power permitted a substantial increase in output of calcium carbide, which was more than double the 1949 level. The total produced was 80,000 tons, against 37,000 in 1949. It was chiefly used for the manufacture of calcium cyanamide.

In the metals sector, Industria Nazionale Alluminio produced 23,000 tons out of a total of 37,000 tons of aluminium produced in the whole of Italy. Production would be expanded to the maximum, and should reach 28,000 tons for the current year.

In the electrical sector, Montecatini had completed the construction of hydroelectric installations at Resia, and in 1950 had increased its available supply of electrical energy by 40% compared with 1949. Consumption of electrical energy by Montecatini group rose to 2,209 million kwh in 1950 from 1,460 million in 1949.

PORTUGAL

New fertiliser factories

Portugal is expected to be self-sufficient in sulphate of ammonia production within a few years. One plant, now almost completed, will start production by the end of the year when, according to present estimates, the hydroelectric schemes now being carried out should supply an adequate amount of power. Output will then be around 50,000 tons p.a. The owners of the plant, the Amoniac Portugal, plan enlargements calling for the investment of a further 80,000,000 escudos.

A second factory is also under construction, for the largest Portuguese industrial concern, the Companhia Uniao Fabril. British specialists are responsible for the installation of this plant, which is expected to be in operation in two years' time.

EGYPT

Fertiliser plant in production

The Suez plant of the Egyptian Fertiliser and Chemical Industries Co. recently came into production and is expected to reach full capacity within two months.

Negotiations for the purchase of equipment to bring natural gas from the oil fields at Ras Ghareb are in progress. The project, when completed, will almost double the plant's capacity.

ISRAEL

Acid plant converted to pyrites

The plant of the Fertilisers and Chemicals Co. Ltd., Haifa, is to be converted at a cost of \$1,000,000 to produce sulphuric acid from iron pyrites by the British method. Up to last month, Israel produced half its sulphuric acid requirements of 30 tons daily, but now production has been severely curtailed and may have to stop completely owing to the lack of elementary sulphur. Many new factories in Israel have been unable to start work owing to the lack of this essential acid.

The halt in production is also affecting local agriculture, which uses one-third of the total sulphuric acid output to produce fertilisers. The Haifa factory is able to produce 20 tons daily when properly supplied with sulphur.

BRITISH GUIANA

Charcoal moisture tests

Experimental equipment is being imported in an effort to trace the reason for the heavy moisture content of local charcoal. If this can be reduced, it is thought that a more favourable market might be found in the U.K.

Bauxite industry expands

The value of bauxite exported for the first 11 months of 1950 showed an increase of over \$1,500,000 compared with the similar period in the previous year. A \$4,000,000 expansion programme has been commenced and will include the installation of additional drying and calcining kilns. Some of the new plant is expected to be in operation in the second half of this year.

AUSTRALIA

I.C.I. expands

Big extensions to nearly all its factories have been planned by Imperial Chemical Industries of Australia and New Zealand, which has a capital of £(A)10,000,000. The plans are based on Australia having a population of more than 11,000,000 by 1960. The company operates three large munition and chemical factories in Victoria, as well as establishments in New South Wales, South Australia and New Zealand.

'I.C.E.' June

This issue will include a special survey of the **Treatment and Disposal of Industrial Waste Waters**, articles on **Sedimentation and Nickel Alloys versus Sulphuric Acid**, and a report of a **Conference on Corrosion**, besides our usual features.

More cement projects

Present cement production in South Australia amounts to 105,000 tons p.a., about 85,000 tons below demand. Production is now to be increased to 230,000 tons p.a. by the end of 1952 in order to meet the State's requirements.

The Adelaide Cement Co. Ltd., is planning extensions to its plant (see *INTERNATIONAL CHEMICAL ENGINEERING*, March 1951, p. 137). A second company, South Australian Portland Cement Co. Ltd., also expects to open a new plant at Port Augusta before the end of 1951.

CANADA

Ammonium-phosphate plant

The Consolidated Mining & Smelting Co. of Canada plans to increase its fertiliser production by building an ammonium-phosphate plant at Kimberly, British Columbia, at an approximate cost of \$9,000,000. The project is part of the company's \$15,000,000 expansion programme in the Province.

The plant at Kimberly will include a unit for processing tailings from one of the company's mines to obtain sulphuric acid, which will be used in treating phosphate rock.

SOUTH AFRICA

Alginic acid to be produced

A factory has been set up in Simonstown to extract alginic acid from seaweed. It will be the first of its kind in Africa. The plant was scheduled to go into operation last month and is expected to export 75% of its output.

The type of seaweed required grows in abundance on South Africa's coast and most of the chemicals needed for the process are also available in the Union. Production costs are expected to be less than in other countries, because artificial indoor drying of the weed, which normally accounts for a substantial portion of production costs, is not necessary in S. Africa where it can be dried out of doors in the sun.

British firm to build battery factory

A £60,000 factory for the production of electrical storage batteries is to be set up at East London, S. Africa. A site of over 4,400 acres has already been purchased by Chloride Electrical Storage Co. S.A. (Pty.), Ltd., producers of Exide batteries. The new plant is expected to be in operation in 18 months' time.

U.S.A.

New synthetic fibre to be made on large scale

The construction of a plant to produce *Amilar* polyester fibre is expected to be started some time this year at the 635-acre site on the Neuse River, near Kington, North California. This fibre has been produced by the Du Pont Co. on a laboratory scale since 1946 under the name of *Fibre V*. It is a condensation polymer obtained from ethylene glycol and terephthalic acid and is not chemically related to nylon, *Orlon* acrylic fibre or any other Du Pont fibre.

Amilar is said to have a high tensile strength and a high resistance to both wet and dry stretching. It has a good resistance to chemical degradation by chemical bleaches and abrasion. Apart from its use for clothing, its industrial applications include fire hose and V-belts.

Plastic tank lining

A new air-drying plastic protective coating has been developed by the U.S. Rubber Co. and is now in pilot-plant production. It is for use in protecting tanks, pipes, fittings, structural steel parts and chemical processing equipment against attack by splash, drip and spray from corrosive chemicals, corrosive atmospheres, weathering and rust, according to the manufacturers.

The plastic is said to combine high film flexibility with good adhesion and impact resistance; it will not chip or crack and can be used on steel, aluminium, concrete, hardwood or composition board. It is applied by spray, allowing 1 hr. drying time between coats and 24 hr. drying time after the final coat. It requires no primer and can be produced in a variety of colours.

MEXICO

New cellulose factory

The new pulp factory reported in *INTERNATIONAL CHEMICAL ENGINEERING*, March 1951, p. 138, is to be located at Chihuahua City instead of at Temsochic. Cellulose, rather than pulp for paper making, will be produced as a result of investigations by Italian technicians, who believe that the ponderosa pine which grows in abundance in Chihuahua, is more suited to the production of cellulose as it has such a low resin content.

The Snia Viscosa of Milan, Italy, will have a financial interest in the project. They will install the plant and lend a number of technicians until production is under way. Considerable state support is also being given.

BRAZIL

Synthetic-nitrogen plants

Plans for the construction of synthetic nitrogen plants are included in projects for development of hydroelectric power in the Sao Francisco Valley.

Meetings

Institution of Chemical Engineers

May 11. 'The Approach to Automatic Process Control,' by A. H. Isaac, 6.30 p.m., Caxton Hall, London, S.W.1.

May 18. Twenty-ninth annual corporate meeting, May Fair Hotel, London.

Society of Chemical Industry

May 7. London Section. 'The Growing of Synthetic Crystals,' by L. A. Dauncey and J. E. Still, 6.30 p.m., London School of Hygiene and Tropical Medicine, Keppel Street, London, W.C.1.

May 8. Chemical Engineering Group. 'Economic Reactor Design,' by G. T. Meiklejohn and R. C. Snell, 5.30 p.m., Burlington House, Piccadilly, London, W.1.

May 10. Road and Building Materials Group. Annual general meeting, followed by 'Pitch and Pitch Mastics as Colloidal Systems,' by Dr. L. J. Wood, 6 p.m., Lecture Hall, Institution of Structural Engineers, 11 Upper Belgrave Street, London, S.W.1.

May 23. Chemical Engineering Group. Thirty-second annual general meeting and dinner, London.

Institute of Petroleum

May 9. Symposium on 'Combustion Reactions in Relation to Gas Turbine Practice,' 5.30 p.m., Manson House, 26 Portland Place, London, W.1.

Fertiliser Society

May 10. Fourth annual general meeting, followed by 'Cliff Quay Works,' by R. J. Parker, and a visit to Cliff Quay works of Fisons Ltd., 11.45 a.m., Ipswich.

Incorporated Plant Engineers

May 10. 'Theory and Practice of Corrosion,' by Dr. Gyngol, Dartford Technical College, and L. C. J. Bayley, Shell Mex Ltd., 7 p.m., Maidstone Technical College, Kent.

May 14. 'Oil.' Talk and film by Scottish Oils and Shell Mex Ltd., 7.30 p.m., Mathers Hotel, Dundee.

May 22-24. Annual conference on plant engineering, Palace Hotel, Buxton.

May 25. 'Laundry Engineering,' by F. Q. Wheatcroft, 7.30 p.m., Imperial Hotel, Birmingham.

May 28. 'Recent Developments in Metallurgy,' by Dr. Irvine, 7.30 p.m., the University, Leeds.

INTERNATIONAL CONFERENCES

June 3-6. Congress on fire-resisting and fire-retarding paints and varnishes, organised by the Fédération d'Associations de Techniciens des Industries des Peintures, Vernis, Emaux et Encres d'Imprimerie de l'Europe Continentale, 28 Rue Saint Dominique, Paris (VIIIe).

June 4-15. Joint engineering conference organised by the Councils of the Institutions of Civil, Mechanical and Electrical Engineers, London.

Recent publications

'Vacuum.' The first issue of a new quarterly journal, *Vacuum*, has been published by W. Edwards & Co. (London), Ltd., manufacturers of vacuum equipment and associated scientific apparatus. It reviews developments in vacuum research and engineering and contains articles by vacuum specialists from all parts of the world as well as comprehensive abstracts of world literature on vacuum. The first issue includes a review of vacuum technology by Prof. E. N. Andrade of the Royal Institution.

Handling of styrene monomer. A safety data sheet on styrene monomer recently published by the Manufacturing Chemists Association Inc., U.S.A., is designed primarily for supervisory personnel and staff members, setting forth information relating to the safe handling and use of the chemical in manufacture, transport and storage, as well as in the laboratory. Specific attention is directed to fire, explosion and health hazards, including personal protective measures and first-aid medical treatment.

Standard autoclaves and reaction vessels. For many years autoclaves and reaction vessels have been designed and manufactured for specific uses in laboratories and pilot-plant work; because of the wide variety of temperatures, pressures and working conditions it is impossible to standardise on one or two designs only. The Kestner Evaporator & Engineering Co. Ltd. have now developed three principal types of autoclave and reaction vessel, namely, direct-heated units, jacketed units and internally heated units. Each of these is available in many different materials of construction and sizes and suitable for a wide range of working temperatures and pressures. A description of this comprehensive range of standard units is given in a leaflet recently published by the company.

Nickel alloys. A comprehensive handbook dealing with the hot-working, annealing and pickling of nickel, *Monel* and *Inconel* has been issued by Henry Wiggin & Co. Ltd. The first section on hot-working deals with suitable fields, hot-working temperatures, methods of forging and with dies and lubrication. The second section covers methods suitable for annealing these high-nickel materials, while the final section gives full details and formulae for pickling.

Industrial electronic timers and relays. The range of timers produced by Elcontrol Ltd. is described briefly in a new data sheet. This range includes an industrial process timer, a delay-on-make timer, a delay-on-break timer, a cyclic interval switch and a weld timer. A second data sheet describes an industrial electronic relay which is claimed to enhance the sensitivity of limit switches, thermostats, dimension gauges, instrument movements and other devices based on limited contact movement and negligible contact pressure.

Coal and non-ferrous metals survey. The two latest contributions to the 'Raw Material Survey' series, published by the Purchasing Officers' Association, deal with coal and non-ferrous metals. That on non-ferrous metals includes details of properties, uses, production and refining of aluminium, copper, lead, tin and zinc, all in short supply today. That on coal includes information on location and reserves, classification, output, production, refining, transport and home consumption.

Vermiculite. The application of vermiculite as a material of construction is described in an illustrated leaflet issued by W. Kenyon and Sons Ltd., whose *Kisol* type is employed for lightweight insulating concrete for roof and floor structures. It also has acoustic insulating and fire-resisting properties. It can also be used for filter aids, absorption media, etc.

High-duty alloy cast irons. *Alloy Metals Review*, published by High Speed Steel Alloys Ltd., Widnes, contains detailed technical information on these metals which indicates their increasingly important role in modern engineering.

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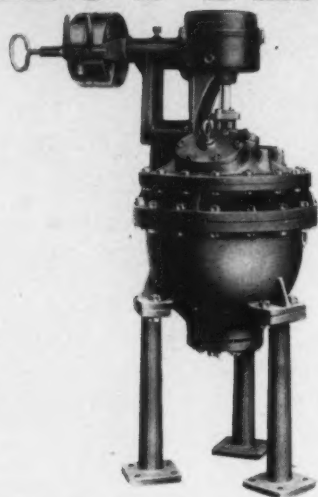


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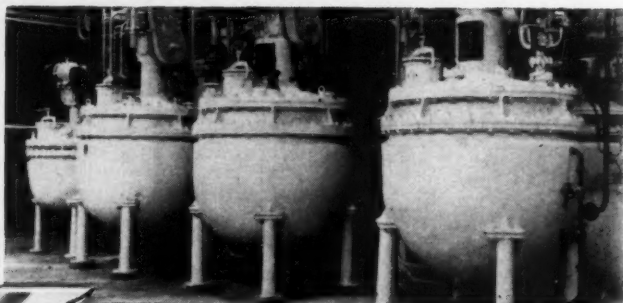


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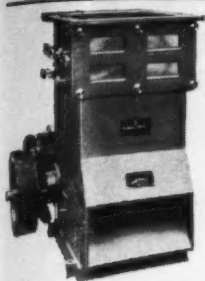
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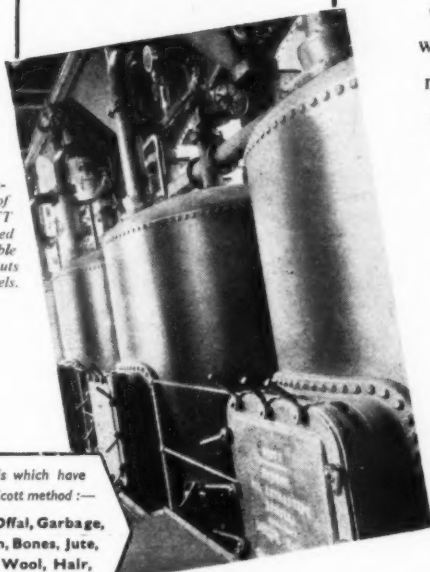
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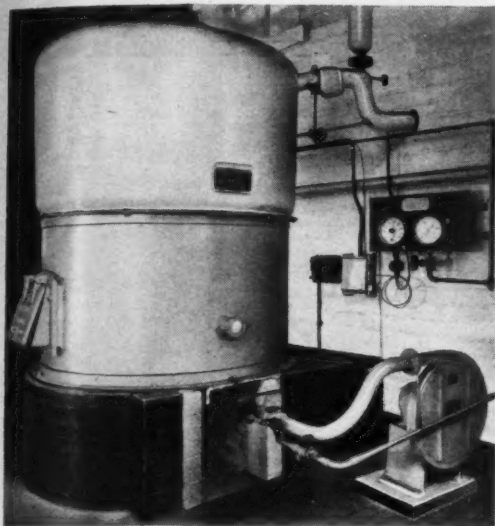


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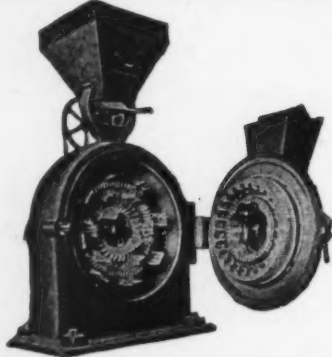
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